

Spontaneous Ignition Characteristics of a Super-Light Oil

by

Abkari Iddris

A Thesis Presented to the

FACULTY OF THE COLLEGE OF GRADUATE STUDIES

KING FAHD UNIVERSITY OF PETROLEUM & MINERALS

DHAHRAN, SAUDI ARABIA

In Partial Fulfillment of the
Requirements for the Degree of

MASTER OF SCIENCE

In

PETROLEUM ENGINEERING

May, 1999

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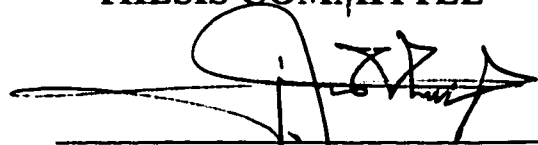
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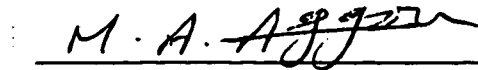
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This thesis, written by **ABUKARI IDDRIS** under the direction of his Thesis Advisor and approved by his Thesis Committee, has been presented to and accepted by the Dean of Graduate Studies, in partial fulfillment of the requirements for the degree of **MASTER OF SCIENCE IN PETROLEUM ENGINEERING**.

THESIS COMMITTEE



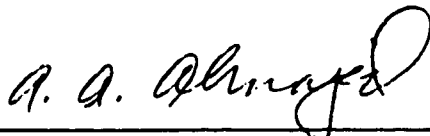
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Dr. Mohamed A. Aggour (Member)



Dr. El-Sayed A. Osman (Member)



Dr. Abdulaziz A. Al-Majed
Chairman, Department of Petroleum Engineering.



Dr. Abdallah M. Al-Shehri
Dean of Graduate Studies



Date: 7/9/99

DEDICATION

To My Parents & To My Family

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ملخص

الاسم : ابو كاري ادريس

عنوان البحث : خواص الاشتعال التلقائي لزيت خام خفيف جدا

التخصص : هندسة البترول

التخرج : مايو ١٩٩٩ م

تم في هذا البحث دراسة خواص الاشتعال التلقائي لزيت خام خفيف جدا ، حيث شُبعت عينات من الرمل بالزيت الخام وأُكسدت عند درجة ١٣٥ مئوية ، وهي درجة حرارة المكنن الذي أُنتج منه الزيت الخام . ولقد تم تغيير بعض العوامل مثل تدفق الهواء ، ونسب التشبع الابتدائية بالزيت والماء ، ودرجة الحرارة ، والضغط الجزئي للأكسجين في تجارب مختلفة لمعرفة أية منظومة من العوامل تسبب في توليد حرارة كافية أثناء الأكسدة لإحداث الاشتعال التلقائي . كما أُجريت إختبارات حرارية باستخدام التحليل الثيرموغرافي والتحليل الحراري التفاضلي لكسب معرفة إضافية عن العمليات التي قد تساهم في إحداث الاشتعال التلقائي .

ولقد فشلت جميع المحاولات في إحداث الإشتعال التلقائي حتى بعد أن تم أكسدة خليط الرمل والزيت بضخ الهواء أو الهواء المخصب بالأكسجين لمدة ست وعشرين ساعة متواصلة . وباللجوء إلى الإختبارات الحرارية أظهرت النتائج أنه بالرغم من أن التأكد منخفض الحرارة يبدأ عند درجة ١٧٠ مئوية ويصل الذروة عند درجة ٢٧٠ مئوية ، بينما يبدأ التأكد مرتفع الحرارة عند درجة ٣٨٠ مئوية ويصل الذروة عند درجة ٤١٥ مئوية ، إلا أن تلك الإختبارات أظهرت مستوى متدن لتوليد الحرارة أثناء التأكد منخفض الحرارة . وتُعزى هذه الظاهرة لقلة نسبة الأسفليات في الزيت . لذا يُستنتج من ذلك أن حقن الهواء في مكنن للزيت الخام الخفيف جدا لا يشكل أية مخاطرة من جهة الإشتعال التلقائي للزيت .

ماجستير العلوم

جامعة الملك فهد للبترول والمعادن ، الظهران ، المملكة العربية السعودية

أيار (مايو) ١٩٩٩ م

THESIS ABSTRACT

FULL NAME OF STUDENT: Abukari Iddris
TITLE OF STUDY : The Spontaneous Ignition Characteristics of a
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The spontaneous ignition characteristics of a super-light oil was investigated. The process involved saturating sand with the crude oil and oxidizing it at a temperature of 135 °C, the temperature of the reservoir from which the oil is produced. Various process parameters such as air flux, initial oil and water saturations, temperature and oxygen partial pressures were varied between experimental runs to determine which set of parameters will cause build-up of enough heat during the oxidation to cause the crude oil/sand mixture to ignite spontaneously. Thermal analysis techniques utilizing Thermogravimetric Analysis (TGA) and Differential Thermal Analysis (DTA) were also used in the study to gain further insight into the processes involved which could possibly lead to spontaneous ignition.

After oxidizing the oil/sand mixture in air or oxygen-enriched air for up to 26 hours continuously, no spontaneous ignition was observed. This is attributed to the inherent lack of asphaltenes in the oil. The results of thermal analysis tests conducted on the crude oil/sand mixture indicated that low-temperature oxidation (LTO) begins at 170 °C and peaks at 270 °C, while high-temperature oxidation (HTO) begins at 380 °C and peaks at 415 °C. Thermal analysis, however, revealed low heat generation during LTO of the oil. Therefore, air injection into the super-light oil reservoir may not pose the risk of spontaneous ignition.

MASTER OF SCIENCE

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CHAPTER 1

INTRODUCTION

When air contacts oil in a reservoir, a low-temperature oxidation (LTO) reaction ensues. This exothermic reaction generates a relatively small amount of heat. If there are no significant heat losses through conduction and convection, the heat build up could cause the sand temperature to rise which could accelerate the reaction into ignition of the oil. This situation is called spontaneous ignition. In field operations, the onset of spontaneous ignition is signaled by two indicators: first is a sudden rise in reservoir temperature with typical temperature levels ranging between 300-600 °F (149-316 °C), and secondly, a drastic drop in produced oxygen[15].

There are occasions when spontaneous ignition can be an asset while on other occasions a liability. For instance, during oil recovery by in-situ combustion, a combustion front is initiated and driven through the reservoir. A critical and essential step in the planning of such a process is the ignition of the oil; therefore spontaneous ignition is not only desired, but it is sometimes promoted by the injection of a highly oxidizable chemical substance into the reservoir. On the other hand, spontaneous ignition is undesirable and becomes a liability during in-situ sand

consolidation of loose formation sand. In this process, LTO is employed to produce a coke-like residue from the oil which cements the sand particles, holding them together. The process however fails if the crude oil ignites spontaneously, initiating a combustion front that burns the sand clean, thus precluding consolidation.

The period of onset of spontaneous ignition can be determined by two methods. One method involves the use of either an analytical or a numerical formulation. These models tend to either under-estimate or over-estimate the time for ignition to occur, depending on the assumptions made during the derivation of these formulations. The second method involves a direct measurement of the ignition time. This is accomplished by means of an experiment using the oil and porous medium of the reservoir to be investigated.

There are several reasons that may warrant investigations into the spontaneous ignition characteristics of crude oil. For instance, when conducted under simulated reservoir conditions in the laboratory, it can verify the possibility of spontaneous ignition in the field. It can also allow investigators to properly select the appropriate ignition methods, equipment and techniques that will greatly assist in achieving a high success rate in field operations. Also, due to their complex nature, crude oils have different oxidation and ignition characteristics[15]. This means that a better understanding of their properties is very essential in order to successfully plan, operate and interpret field ignition operations. Furthermore, since ignition could take a considerably long time to achieve in field operations,

an estimate of its delay time will assist operators in deciding which of the two means (artificial or spontaneous) is more economic.

For this study, investigating the spontaneous ignition characteristics of the crude oil under study is very essential for the sand consolidation project proposed for the field. More details on this aspect of the study are provided in the next chapter.

CHAPTER 2

STATEMENT OF THE PROBLEM AND OBJECTIVE OF THE STUDY

2.1 STATEMENT OF THE PROBLEM

A field in central Saudi Arabia which produces a super-light oil, of 52 degree API, has a serious sand production problem and has, therefore, become a target of research on a new sand consolidation technique. This new method involves the use of LTO processes to generate a coke-like residue to bind the sand grains. However, experience has shown that continued oxidation of oil-saturated sand can cause the oil to ignite spontaneously, thus rendering consolidation impossible. Therefore, investigation of conditions that can lead to spontaneous ignition of the oil during any in-situ sand consolidation by LTO becomes imperative; which therefore brings us to the objective of this study.

2.2 OBJECTIVE OF THE STUDY

The main objective of this study is to explore the spontaneous ignition potential and characteristics of the super-light crude oil from central Saudi Arabia. Specifically, the ignition characteristics are to be correlated with the reservoir and process conditions.

Before starting an investigation of this nature, it is essential to examine the various literature works that are relevant to the study. These works are reviewed in the next chapter.

CHAPTER 3

LITERATURE REVIEW

LTO reactions are known to be the major causes of spontaneous ignition. These reactions occur when oxygen reacts with the crude oil at relatively low temperatures, usually about 400 °F (204 °C), producing mostly water and oxygenated hydrocarbons such as alcohols, aldehydes, ketones, carboxylic acids and hydroperoxides[14]. Some of the major observations made in the literature on LTO studies are listed as follows (1,3,9,10,11,16):

- (i) When an oil/sand mixture is subjected to LTO reactions for an extended period of time, deposition of coke-like residue on the sand grains results. The extent of this deposition is dependent on the API gravity of the crude as well as the oxidation temperature.
- (ii) Measured oxidation rates for different crude oils indicate that light crudes are more susceptible to partial oxidation at low temperatures (LTO) because of their relatively high hydrogen content.

- (iii) LTO reactions cause spontaneous ignition to occur. The process is catalyzed by the presence of clay and certain metallic derivatives either in the sand or the oil.
- (iv) LTO reactions significantly increase the asphaltene content of the oxidized oil. Asphaltene content, density and viscosity are well correlated with the extent of oxidation.

Since spontaneous ignition is the subject of investigation, a review of it's relevant literature is presented below.

In 1958, Tadema and Quant:[16] in their patented work, provided a method of ignition which will guarantee a uniform combustion of the oil in the formation. To achieve this, their method proposed the injection of a highly oxidizable liquid, together with or followed by a gaseous oxidant, into the formation to promote spontaneous ignition. The choice of gaseous oxidant should be such that it reacts spontaneously with the liquid under operating reservoir conditions of temperature and pressure. After ignition, air is used to maintain the actual combustion that follows. In the case of a formation in which high temperature and/or high pressure naturally prevails, the method further suggests that the readily oxidizable liquid may be omitted entirely, provided an oxygen-rich gas (or even pure oxygen) is chosen as the oxidant. Warren et al[36], referring to the possibility of spontaneous ignition, stated that at a finite absolute temperature, a reaction

rate exists; hence the injection of air into a partially oxidized oil is likely to cause ignition to occur spontaneously.

After several unsuccessful attempts to spontaneously ignite crude oil/sand mixture in a combustion tube, Caruthers[8], in 1965, observed that at temperatures much lower than the ranges he studied, 400-472 °F (204-246 °C) (petroleum reservoirs have much lower temperatures than this range) it will be necessary for the combustion tube to be longer than 21 inches, or an oxidizing agent other than air be used, to enable one to observe ignition. After eventually being able to achieve spontaneous ignition at high temperatures, he concluded that the location of spontaneous ignition in a porous medium is highly dependent upon the air injection rate and the initial temperature. The lower the initial temperature, the greater the amount of air required to induce ignition at the same point within the tube, also, ignition will occur farther from the inlet at constant air flux.

In 1970, Tadema and Weijdemans[18] developed an analytical formulation for estimating the time that spontaneous ignition would occur. The model however under-estimated the ignition delay time because of neglect of heat loss considerations. By utilizing kinetic data obtained from measured oxidation rates of crude oil at low temperature to test their formulations, they found that neither the sand grain size (in the permeability range of 0.3 – 28 darcies) nor the oil/sand ratio (92-12% by weight of sand) appeared to have any significant influence on the oxidation rate. They also concluded that prolonged oxidation did not seem to affect the chemical reactivity of the crude oil sands.

In an attempt to improve on the analytical model described above, Burger[4], in 1976, developed a numerical simulator to investigate the spontaneous ignition characteristics (ignition delay and position) of oil in porous media. The proposed model considered both radial and longitudinal flow geometries and also accounted for heat transport by conduction and convection. Other than estimating a longer ignition time than that of Tadema and Weijdemans[18], the study showed that during the ignition delay, the maximum temperature in the heated zone increases exponentially with time. Also, the distance from the injection well to the ignition zone increased with the air flow rate and with the delay time.

However, Prats[14] observed that Burger's [4] calculated ignition times are only a few percentage longer than that predicted by the analytical expression of Tadema and Weijdemans[18], except for reservoirs at low initial temperatures, where the difference could be very large.

Howard et al[12] , reported in their field studies that the effect of cooling by injected air caused ignition to occur at a distance of between 50 to 110 feet from the injection well.

From the literature just examined, it can be seen that there are two general methods of investigating spontaneous ignition characteristics of oil: one is by use of formulations (analytic or numerical models) and the other is by direct measurement through experimental means. While the first method might be economical and used when experimentally determined parameters are unavailable, the second method is usually the most

preferred because of its close representation of what happens in the reservoir. Therefore, for this investigation, the direct means by experimental study is chosen.

CHAPTER 4

EXPERIMENTAL WORK USING OXIDATION REACTORS

4.1 (a) Oxidation Apparatus

Figure 4.1 shows the setup used in this investigation. It consists of three basic components: the reactor and its associated connections, gas supply systems and measuring and storage systems. A total of five reactors of different sizes were used. Table 4.1 shows the detail dimensions of the reactors. Reactor #1, 3 and 4 are made from stainless steal; Reactor #2 is a copper slim tube that was wound into a spiral coil to fit inside the heating oven, while Reactor #5, the largest reactor, is made from galvanized steel. Some of the reactors have flanges at their tops and bottoms while the others have a compression fitting fitted to provide entry point to the thermocouples. Figure 4.2 shows a typical flanged reactor. On the upstream part of the reactor is the gas supply system consisting of compressed cylinders of air, nitrogen and a mixture of the two, connected to 'Drierite' drying beds and gas measuring and flow control devices. The downstream portion of the reactor is connected to the temperature measuring thermocouples read-out as well as gas analyzer.

Two gas analyzers were used in this study: The first was a 'Kane-May' model KM9004 Electronic Combustion Gas Analyzer, used with Reactor #1. It measures the oxygen, carbon dioxide and carbon monoxide content of the effluent gas. It was sensitive to the pressure of the incoming gas and had a low accuracy. This analyzer was later replaced by the second analyzer, a 'Rosemount' model 755A Analytical Oxygen Analyzer. This had a higher accuracy and gives the oxygen content of the effluent in the range of 0-100.00%. It has a scale expansion and zero- suppression mechanism for calibration to any desired span.

Spontaneous ignition was monitored by the thermocouple reader which provided sand face temperature, and the oxygen analyzer which provided the oxygen concentration in the effluent gas. These two parameters are recorded with time by the data acquisition system. The nature of the crude oil and porous media used in the study are described below.

LEGEND

- | | | |
|-------------------------|-----------------------------|-----------------------------|
| 1. Pressure Gauge | 7. Pre-Heating Coil | 13. Acid Scrubber |
| 2. Valve | 8. Reactor | 14. 2-Micron Filter |
| 3. Two-Way Valve | 9. Thermowell | 15. Mass Flow Meter |
| 4. 'Drierite' Bed | 10. Thermocouple | 16. Oxygen Analyzer |
| 5. Mass Flow Controller | 11. Ice Bath | 17. Thermocouple Reader |
| 6. Oven | 12. Back Pressure Regulator | 18. Data Acquisition System |

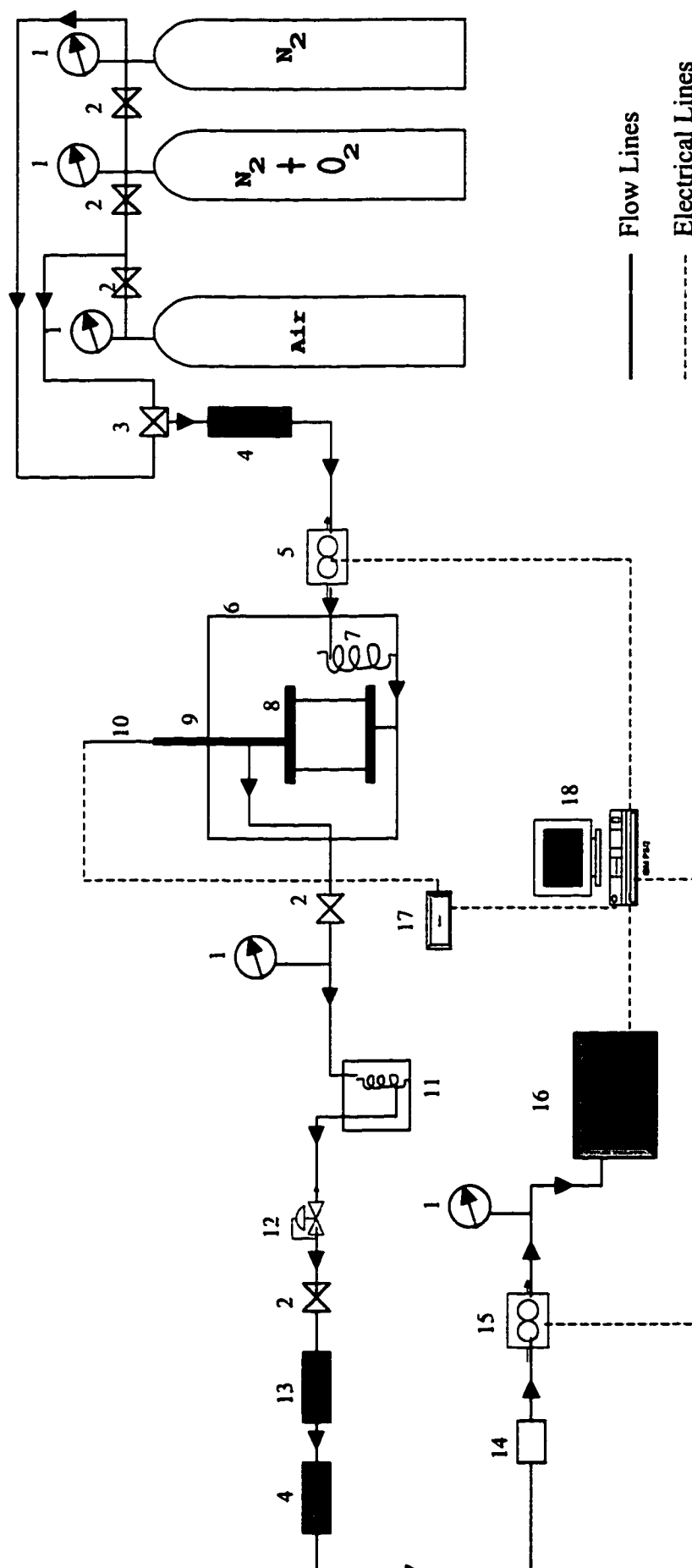


Figure 4.1: Low Temperature Oxidation Apparatus

Table 4.1: Reactors Used in the Experiment

| REACTOR | DIMENSIONS (Inches) | | |
|-------------|---------------------|---------------------|--------------------|
| | LENGTH | OUTSIDE DIAMETER | INSIDE DIAMETER |
| Reactor # 1 | 4.00 | 1.31 | 1.00 |
| Reactor # 2 | 148.9 | 0.50 | 0.45 |
| Reactor # 3 | 15.75 | 0.75 | 0.47 |
| Reactor # 4 | 14.00 | 1.88 | 1.62 |
| Reactor # 5 | 11.38 | 4.44 | 4.19 |

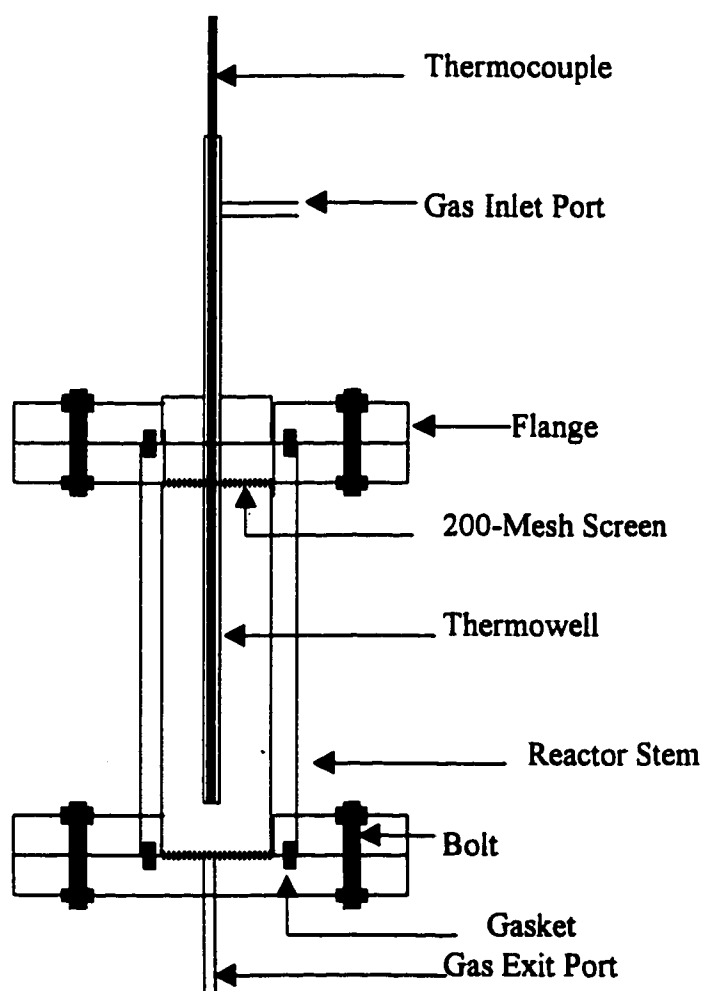


Figure 4.2: Typical Reactor Assembly as used with the Flanged Stainless Steel and Galvanized Steel Large Tubes

4.1 (b) Experimental Material

Oil # 1, the 52 degree API-gravity crude was the main oil of this investigation. However, a blend of Oil # 1 and 2 was also used to see the effect of increased asphaltene content on the rate of oxidation. Oil # 2 is an 18 degree API-gravity heavy crude from the eastern province of Saudi Arabia while Oil # 3, although from the same province, is a medium crude of 22 degree API gravity. A brine made of 4% Potassium Chloride (KCL) solution was also used in experimental runs where residual water saturation was desired. Table 4.2 shows the properties of the oils and brine used.

Two porous media were used. The first was a field produced sand that was recovered from a field separator during routine clean up operations. This was the field from which Oil # 1 was produced. This sand had some clay in it. The second sand was obtained from an outcrop of Biyadh formation in central Saudi Arabia. It was clean and white and had no clay. Sieve analysis of the two sands, shown on Table 4.3 and Figure 4.3, indicate that both sands were well sorted. In most of the runs, the first sand was used.

Table 4.2: Properties of Oil and Brine

| FLUID TYPE | API-GRAVITY AT 60 °F | VISCOSITY (cp) AT 24 °C | DENSITY (g/cc) AT 24 °C | SATURATES (wt. %) | AROMATICS (wt. %) | POLARS (wt. %) | ASPHATENES (wt. %) |
|------------|-------------------------|-------------------------------|-------------------------------|----------------------|----------------------|-------------------|-----------------------|
| Oil #1 | 51.1 | 1.769 | 0.7786 | 80.064 | 19.470 | 0.286 | 0.180 |
| Oil # 2 | 17.9 | .6503 | 0.9470 | 13.22 | 54.24 | 28.44 | 4.10 |
| Oil # 3 | 22.2 | 93.284 | 0.9111 | - | - | - | - |
| Brine | - | 0.852 | 1.004 | - | - | - | - |

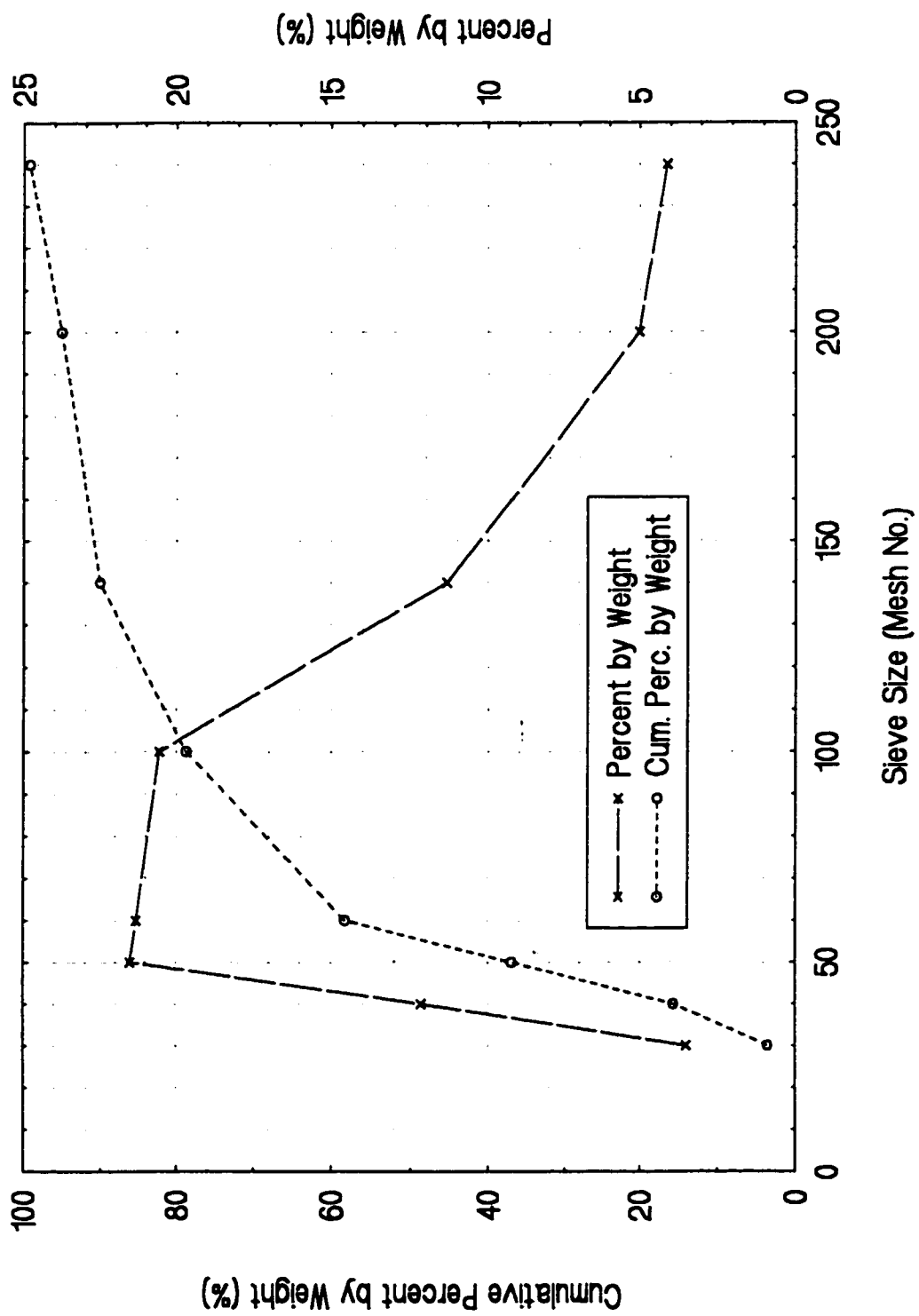


Figure 4.3(a): Sieve Analysis of Field Produced Sand

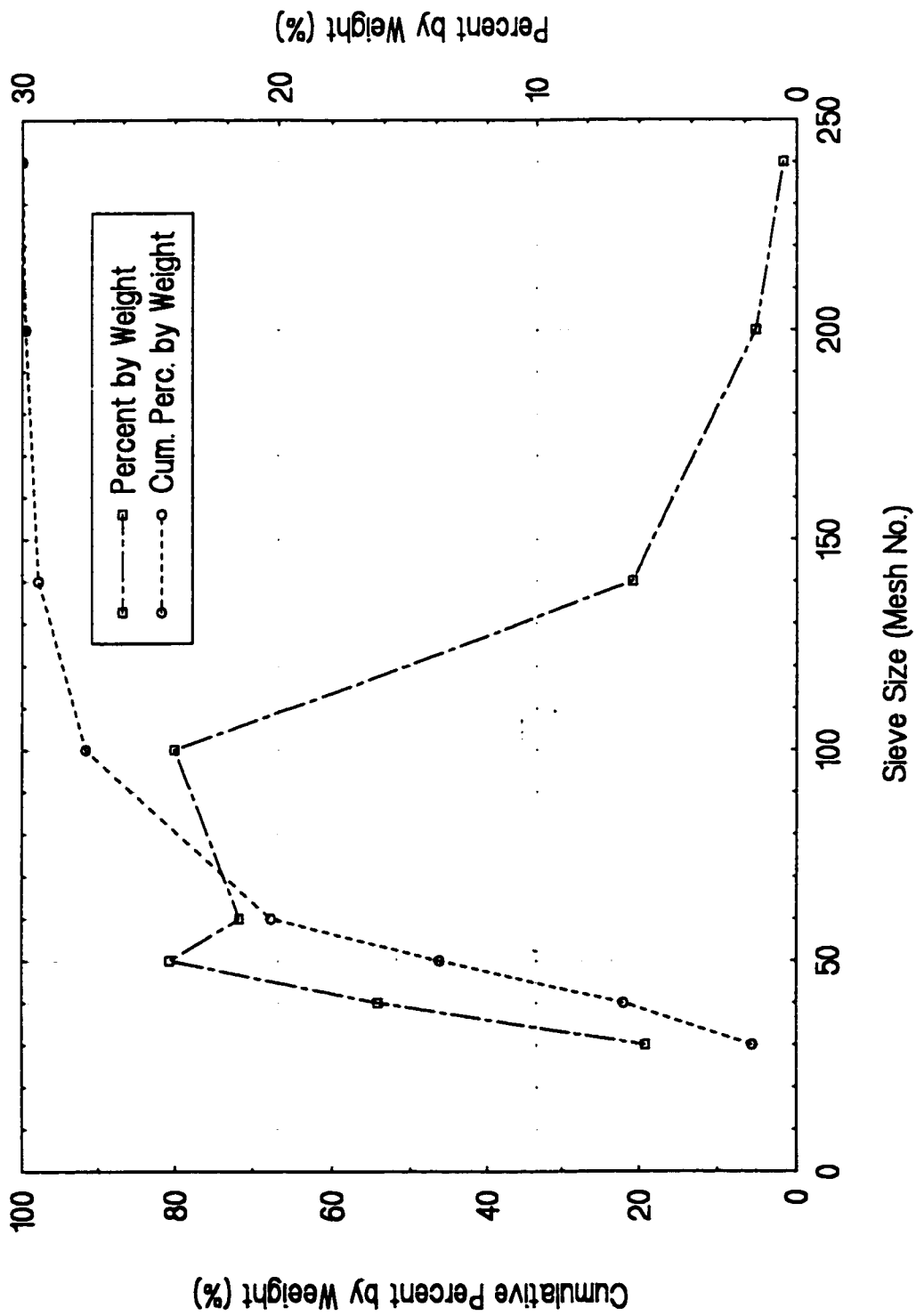


Figure 4.3(b): Sieve Analysis of White Sand

Table 4.3 (a): Sieve Analysis of Field Produced Sand

| SIEVE SIZE (MESH NO.) | WEIGHT RETAINED (gm) | PERCENT BY WEIGHT (%) | CUMULATIVE PERCENT BY WEIGHT (%) |
|----------------------------------|-------------------------------------|--------------------------------------|---|
| 30 | 34.8 | 3.48 | 3.48 |
| 40 | 121.4 | 12.14 | 15.62 |
| 50 | 215.3 | 21.53 | 36.93 |
| 60 | 213.1 | 21.31 | 58.24 |
| 100 | 205.5 | 20.55 | 78.79 |
| 140 | 113.4 | 11.34 | 90.13 |
| 200 | 50.1 | 5.01 | 95.14 |
| PASSED 200- (PAN) | 41.2 | 4.12 | 99.26 |

Table 4.3 (b): Sieve Analysis of White Sand.

| SIEVE SIZE (MESH NO.) | WEIGHT RETAINED (gm) | PERCENT BY WEIGHT (%) | CUMULATIVE PERCENT BY WEIGHT (%) |
|----------------------------------|-------------------------------------|--------------------------------------|---|
| 30 | 54.9 | 5.58 | 5.58 |
| 40 | 160.2 | 16.23 | 21.81 |
| 50 | 256.0 | 26.03 | 47.84 |
| 60 | 223.3 | 22.70 | 70.54 |
| 100 | 219.3 | 22.30 | 92.84 |
| 140 | 52.5 | 5.34 | 98.18 |
| 200 | 13.3 | 1.35 | 99.53 |
| PASSED 200- (PAN) | 4.0 | 0.41 | 99.94 |

4.1 (c) Experimental Procedure

A typical run was started by first filling the reactor with the porous medium and saturating it with brine (for runs designed to have residual water saturation). This was then followed by saturation with crude oil. Finally, displacement by nitrogen followed at a very low pressure to create a gas phase permeability in the sand and at the same time displace the mobile oil. With the largest reactor, where saturation by injection was impractical, the sand/oil mixture was prepared in a plastic container and tamped into the reactor. In all cases, the initial oil, water and gas saturations were determined through mass balance. The assembled reactor was then placed vertically in the oven and pressure-tested with nitrogen. Nitrogen flow, from the bottom of the reactor to the top, is maintained throughout the heating period so as to avoid premature oxidation. When the desired temperature of about 135 °C (the temperature of the reservoir from which Oil # 1 was obtained) is reached, nitrogen flow is discontinued and the oxidant (air or gas mixture) is initiated at the specified flow rate, system pressure and temperature. These conditions are maintained constant throughout a run, but varied from one run to the other. Continued recording of the sand pack temperature, mass flow of produced and injected gases, the oxygen content of the produced gases are all monitored and stored by the data acquisition system. A run is terminated when ignition is detected or when the oxygen content of produced gas and the temperature of the sand pack remained constant for a sufficiently long period of time. The ranges of initial and operating conditions of all oxidation runs were as follows:

- ◆ Oxidation Temperature Range = 100 – 174 °C
- ◆ Average System Pressure Range = 80 – 1, 050 Psig.
- ◆ Initial Oil Saturation Range = 14 – 81 % P.V.
- ◆ Initial Water Saturation Range = 20.4 – 33.8 % P.V.
- ◆ Air Injection Time = 3 – 26 Hours
- ◆ Gas Injection Rate = 36 - 300 cc/min.
- ◆ Total Number of Reactors Used = 5

4.2 RESULTS AND DISCUSSIONS OF OXIDAITON RUNS

A total of 32 oxidation runs were conducted out of which 23 shall be reported and discussed below. The remaining runs not reported were either the ones that were initially conducted for the purpose of establishing a procedure for the study or were among the ones that were aborted due to technical reasons.

4.2.1 Oxidation Results from Reactor # 1.

Table 4.4 shows the first set of reported experimental results for the six runs with Reactor # 1, the stainless steel flanged reactor. As the parameters were changed from one run to the other, their effect on spontaneous ignition was determined by observing the rise in sand temperature and also the oxygen concentration of the exit gas.

All six runs showed only a meager rise in sand pack temperature. The highest rise in temperature was 3 °C in Run 06-FR while the rest showed only a degree or two rise in temperature. Within the accuracy of the combustion gas analyzer, very low changes in oxygen concentration was observed during the oxidation process in almost all the runs. Runs 01-FR to 04-FR showed no change in oxygen concentration between the injected air and the exit gas, while runs 05-FR and 06-FR showed a drop of 1.9% and 2.0% in oxygen content, respectively. No carbon oxides were detected in the exit gas. The duration of most experimental runs ranged between 6-8 hours with the exception of the first run, which was conducted for 26 hours. Also, at the end of each run, the partially oxidized sand was recovered from each reactor and carefully examined. Each time, the sand was found to be soft in texture, light brownish in color and wet with a hydrocarbon odor.

All six runs described above did not show any characteristics of spontaneous ignition after between 8 to 26 hours of oxidation. In an attempt to enhance the LTO reactions, the reactors in all runs subsequent to run 01-FR were insulated while in the oven. This measure was intended to maintain adiabatic conditions and avoid the dissipation of heat generated by the oxidation reaction. The air flux was also reduced with the hope of reducing heat loss by convection. This is because at high air injection rates, any heat generated by the oxidation reaction can be carried away by the incoming stream of air just as fast as the heat is generated. Reducing the air injection rate also increases the oxygen residence time within the reactor so as to enhance efficient oxidation.

Table 4.4: Results of Low Temperature Oxidation Using Reactor # 1.

| RUN NO. | INITIAL OIL SATURATION OF SAND PACK (%PV) | INITIAL REACTOR TEMPERATURE (°C) | INITIAL REACTOR PRESSURE (PSIG) | AIR INJECTION RATE (CC/MIN.) | RESULTS/OBSERVATION |
|---------|---|----------------------------------|---------------------------------|------------------------------|--|
| 01-FR | 32.8 | 101 | 100... | 300 | <ul style="list-style-type: none"> - No ignition observed after 26 hours. - Sand color slightly brownish , no hydrocarbon odor - Temperature rise = 2 °C - No change in oxygen concentration |
| 02-FR | 42.6 (Swi=20.3) | 102 | 100 | 36 | <ul style="list-style-type: none"> - No ignition observed after 6 hours. - No change in temp. and oxygen levels |
| 03-FR | 38.4 | 174 | 80 | 36 | <ul style="list-style-type: none"> - No ignition observed after 8 hours. - No change in temp. and oxygen levels |
| 04-FR | 33.0 | 144 | 100 | 50-36 | <ul style="list-style-type: none"> - No ignition observed after 8 hours. - No change in temp. and oxygen levels |
| 05-FR | 24.6 | 149 | 100 | 50-38 | <ul style="list-style-type: none"> - No ignition observed after 8 hours. - Oxygen concentration dropped by 1.9 % |
| 06-FR | 29.4 | 150 | 80 | 294 | <ul style="list-style-type: none"> - No ignition observed after 8.5 hours. - Temperature rise = 3 °C - Oxygen concentration dropped by 2 % |

In run number 03-FR, the initial oxidation temperature was increased to 174 °C while the air injection rate was reduced to 36 cc/min. In spite of these changes, no indication of spontaneous ignition was observed.

Since no spontaneous ignition was achieved with the first reactor, it became necessary to make some changes in the set up, so as to improve any chances of spontaneous ignition if it occurs. One such change was the replacement of the less-sensitive Combustion Gas Analyzer with the more accurate Analytical Gas Analyzer, which was attached to the data acquisition system for direct monitoring of the changes in the operating parameters. However, since the analyzer only measures oxygen concentration, carbon dioxide and carbon monoxide concentration data were no longer available. Presented in a tabular form in the appendix are the results of all experimental runs recorded by the data acquisition system. The data is corrected to account for interruptions during the run. The first column is the actual clock time in hours. The second column is the temperature of the sand pack at the thermocouple point of contact and the last column is that of the oxygen concentration in the effluent gas stream.

4.2.2 LTO Results from Reactor # 2

Reactor # 2 is a 12-foot long copper tube of 0.5 inch O.D. with a wall thickness of 0.032 inches. The idea was to increase the oxygen residence time in the pack and lessen the heat loss across the insulated thin wall.

The first run, 01-ST, lasted for 8 hours without any indication of ignition. In the second run, run # 02-ST, a blend consisting of 10% by volume Oil # 2 and 90% by volume Oil # 1 was used. Oil # 2 has a high asphaltene content (4.1% by weight) as shown in Table 4.2. Figure 4.4 shows the LTO profile of this run. During the first two and half hours of air injection, there was an increase in temperature by 3 °C (from 138 to 141 °C) while the oxygen concentration in the effluent gas dropped by about 11%. These trends are characteristic of the eminence of spontaneous ignition, which was probably enhanced by the high asphaltene content of the blend. However, after about three hours of oxidation, the trends began to reverse. This is seen by the sudden reduction in temperature and an increase in oxygen concentration of the effluent gas.

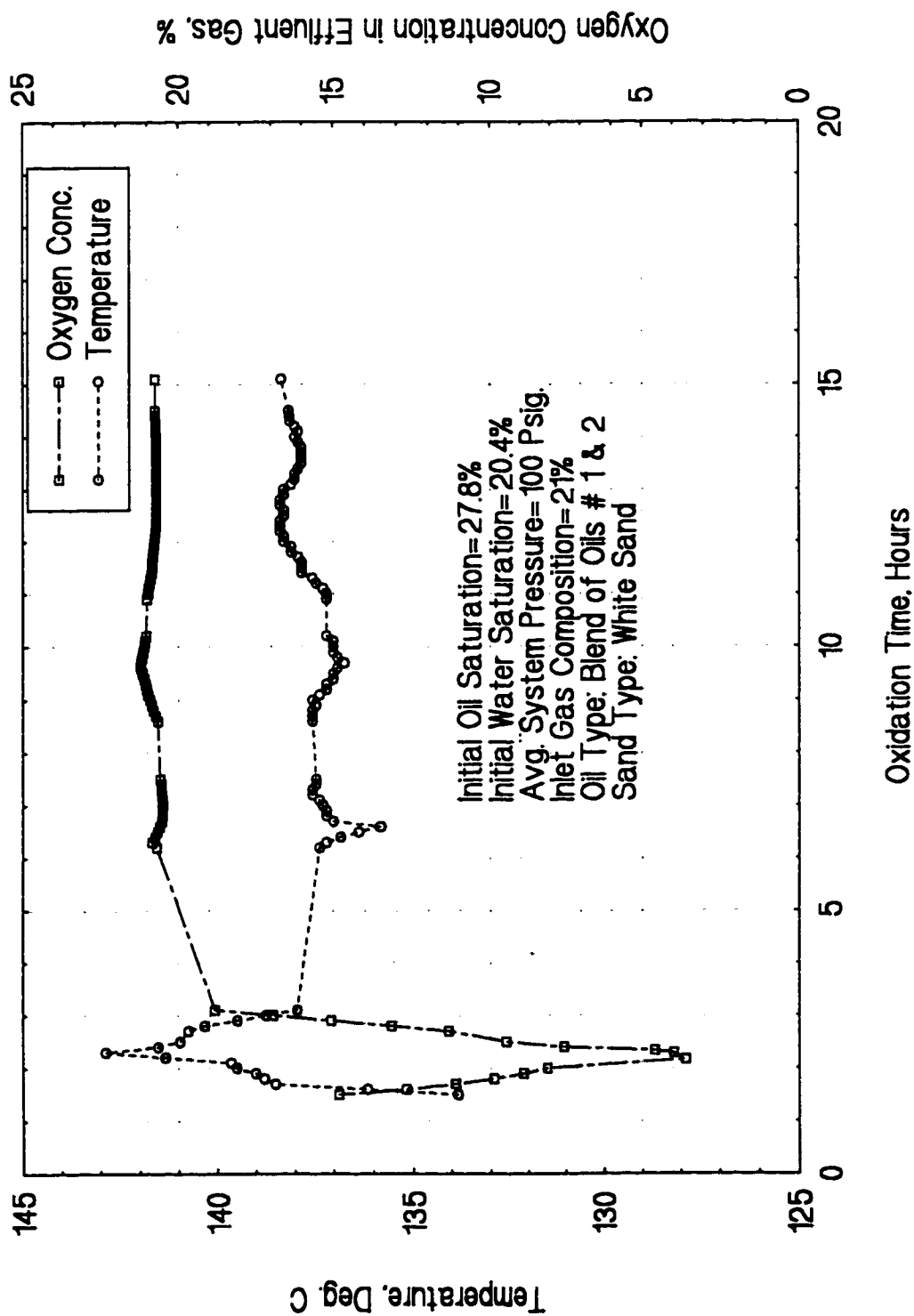


Figure 4.4: Low Temperature Oxidation Profile for Run # 02-ST

As a means of enhancing a higher chance of observing spontaneous ignition, a reactor that could be efficiently insulated was built. This was the 12-inch long stainless-steel reactor, Reactor #3. The results are discussed below.

4.2.3 Oxidation Results from Reactor #3

During the first run, Run # 01-SST, the sand pack was initially saturated with brine (4% KCL) and the brine displaced by Oil #1. Thereafter, both brine and oil were displaced by nitrogen gas to residual levels. The initial fluid saturations prior to oxidation were 58.9% oil and 33.8% brine. The idea was to see what effect, if any, the presence of residual water saturation had on the rate of oxidation and ultimately spontaneous ignition. After nearly 9 hours of continuous air injection, as shown in Figure 4.5, there was a rise in temperature by only 3 °C and a drop in oxygen concentration in the effluent by 0.18%. This showed that initial water saturation did not have any effect on the rate of oxidation at the temperatures investigated. During the next four runs, seen in Figure 4.6 through Figure 4.9, reduction in air flux did not significantly improve any rise in temperature or drop in oxygen concentration in the effluent gas.

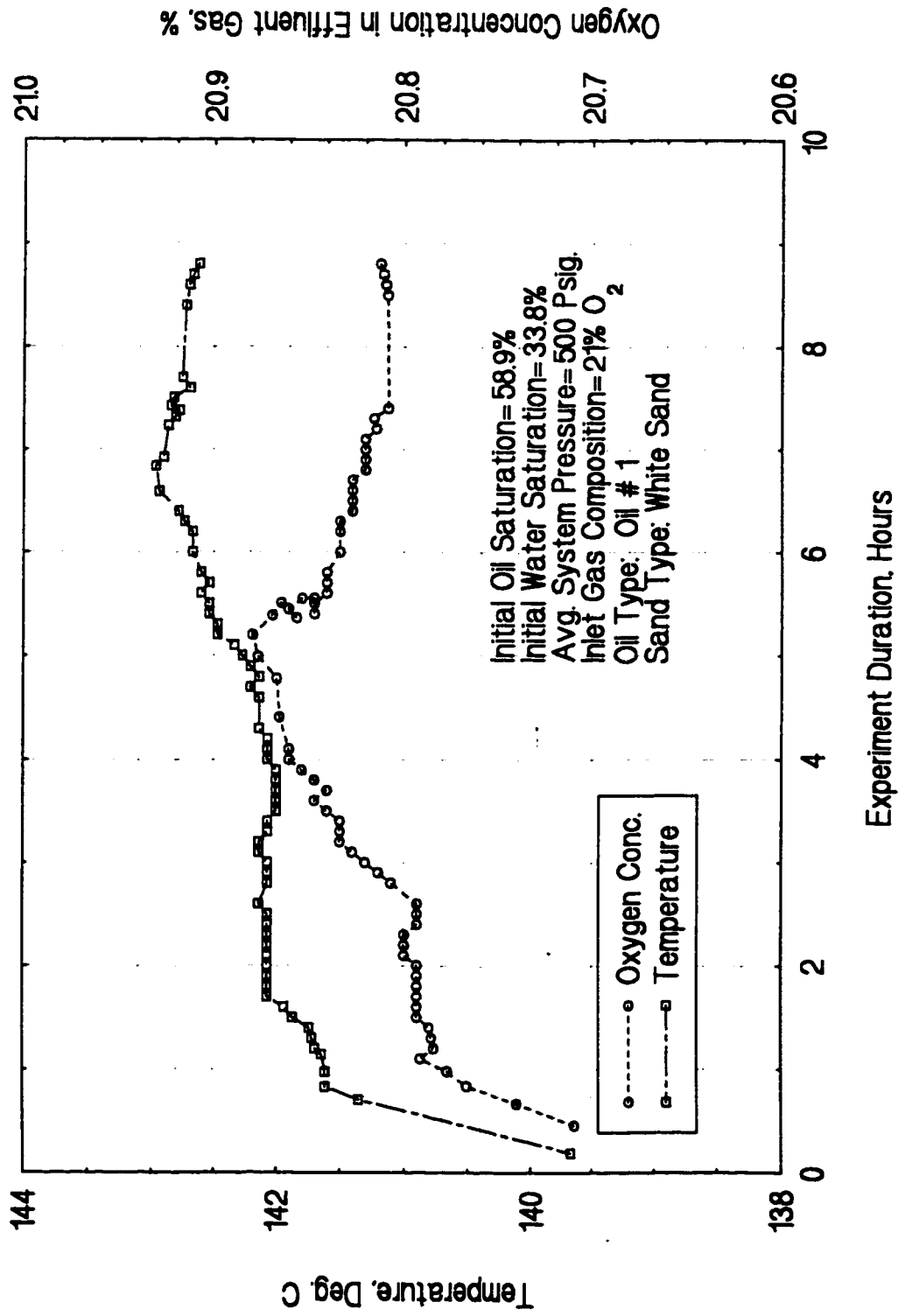


Figure 4.5: Low Temperature Oxidation Profile for Run # 01-SST

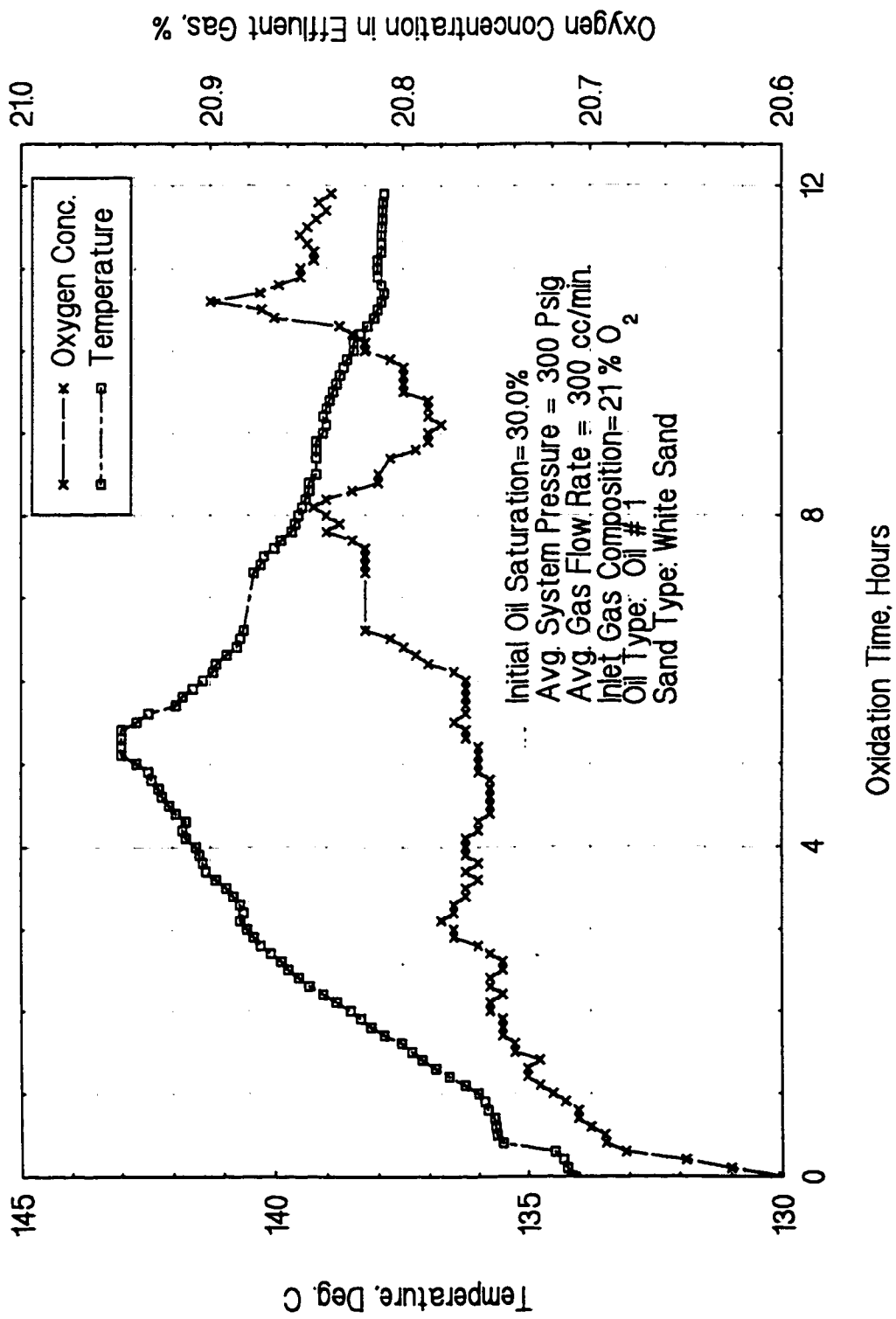


Figure 4.6: Low Temperature Oxidation Profile for Run # 02-SST

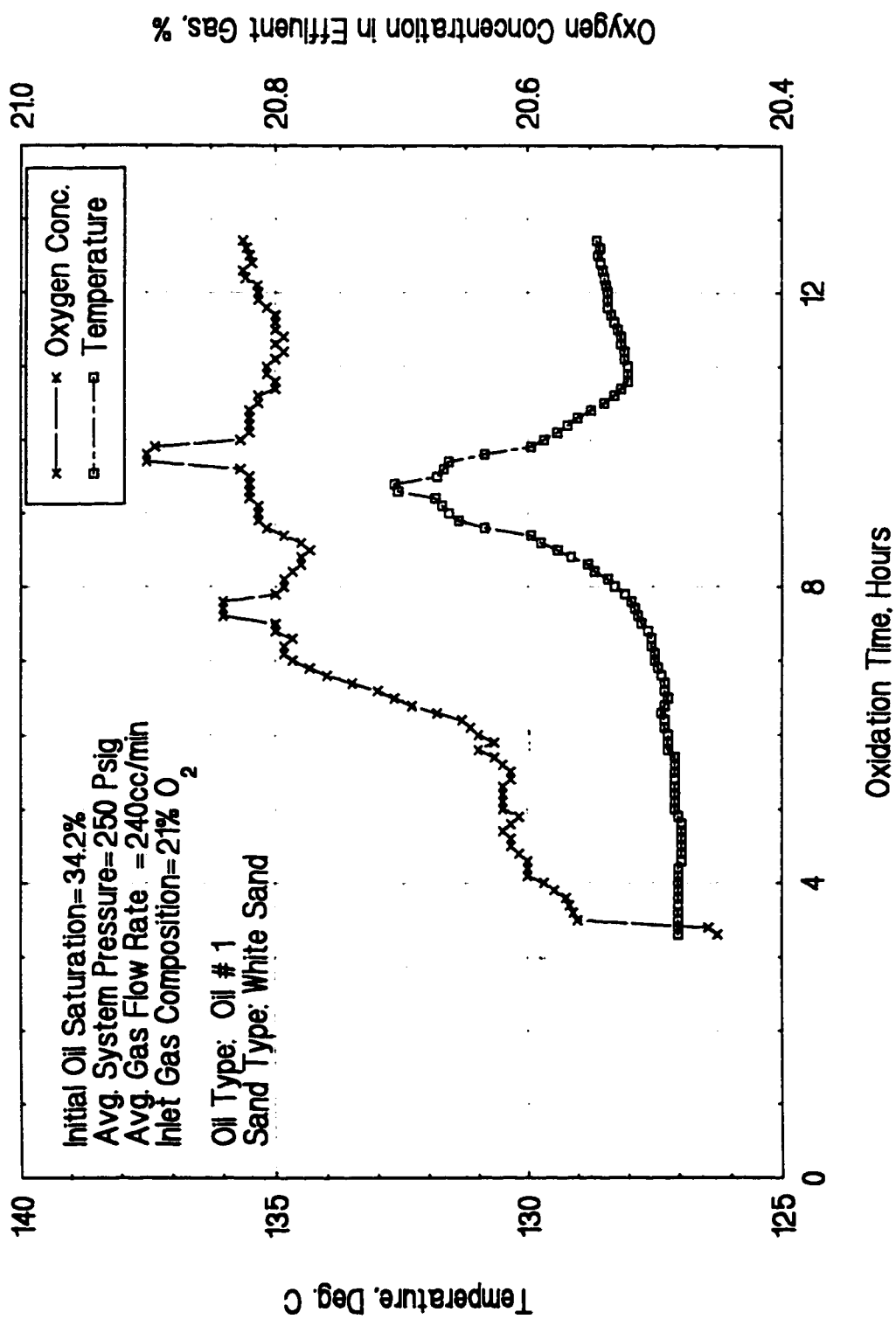


Figure 4.7: Low Temperature Oxidation Profile for Run # 03-SST

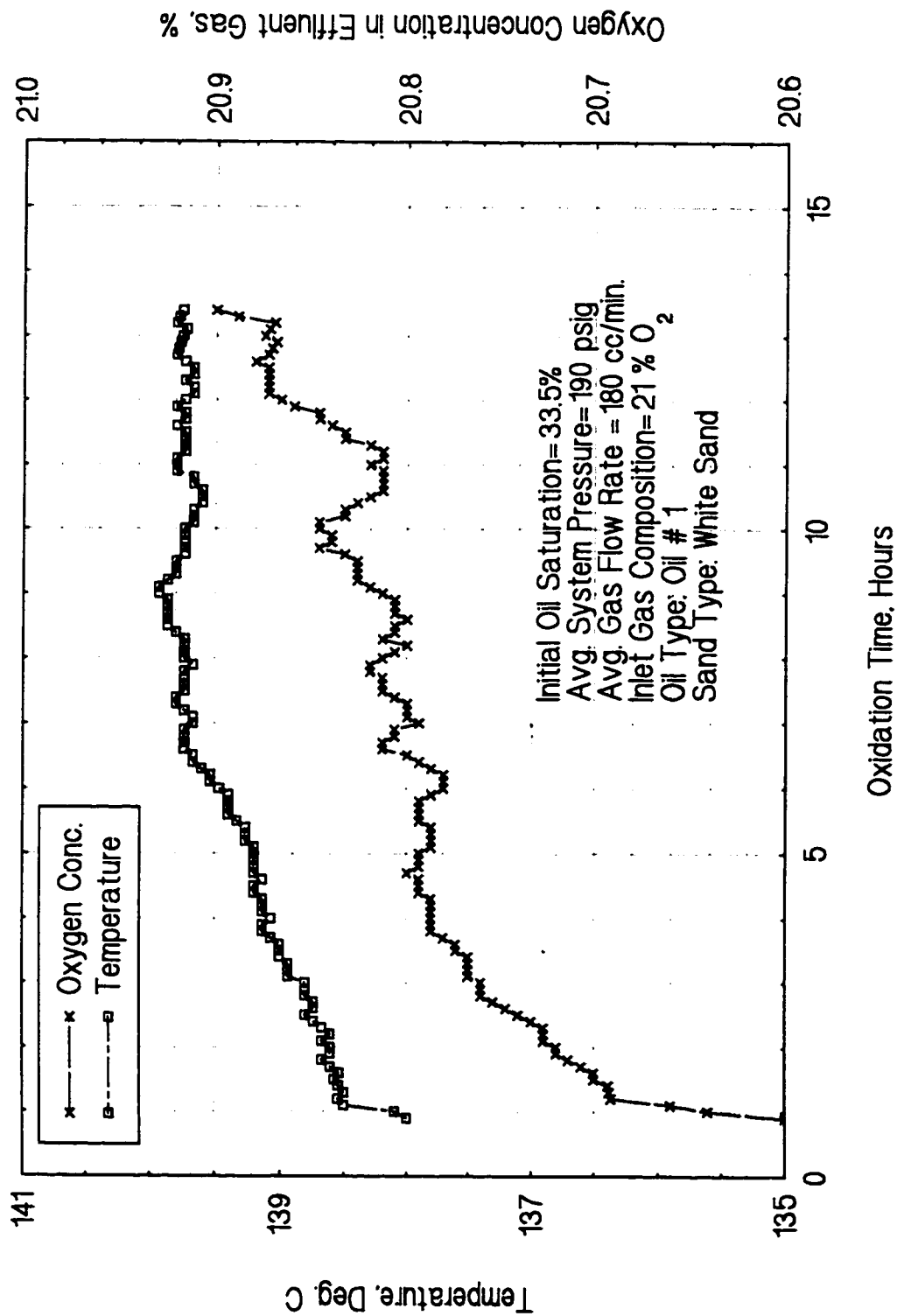


Figure 4.8: Low Temperature Oxidation Profile for Run # 04-SST

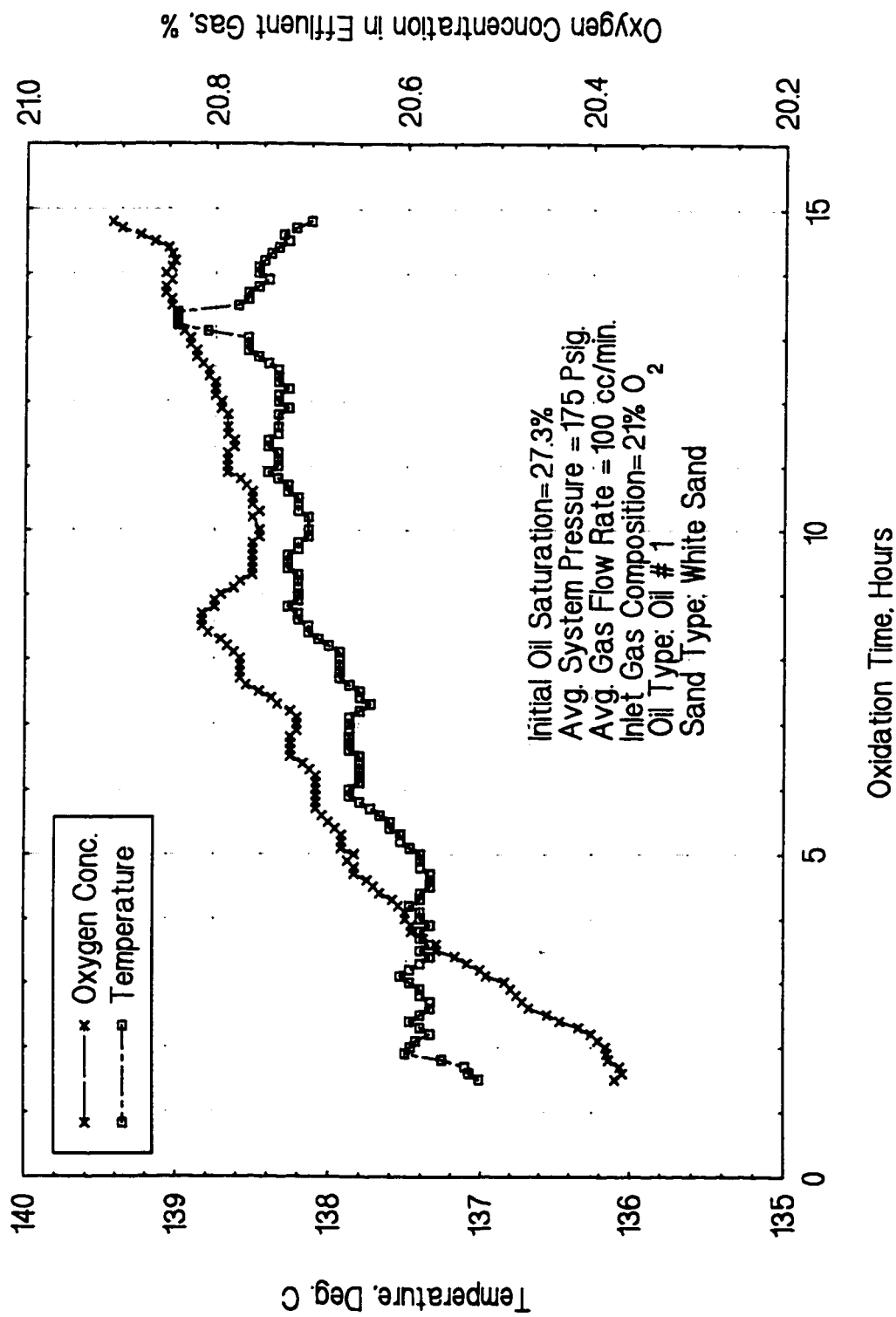


Figure 4.9: Low Temperature Oxidation Profile for Run # 05-SST

At this point in the investigation, the oxidant was replaced by enriched air that had 40 % oxygen. This was to increase the rate of LTO reactions. Initial oil saturation, system pressure and gas injection rate were also adjusted with the hope of improving any chance for ignition to occur. Effects of these changes are reflected in the oxidation profiles of the next three runs, Figures 4.10 to 4.12. As can be observed, there was no visible indication of spontaneous ignition. Other than a couple or so degrees in temperature increment, the curves remained unchanged for several hours.

4.2.4 Oxidation Results from Reactor #4

To further enhance the chances of spontaneous ignition, a still larger reactor was built while running the experiment at much higher pressures using the oxygen-enriched air. A larger reactor would contain a larger mass of porous medium that will provide effective adiabatic conditions. As can be seen from Figures 4.13 and 4.14, runs with Reactor # 4 did not show any significant rise in temperature or drop in oxygen concentration, despite the high initial oil saturation of up to 56.2 %. As before, slight changes in temperature and oxygen concentration in the effluent gas leveled off to almost constant values for twelve to fifteen hours of air injection.

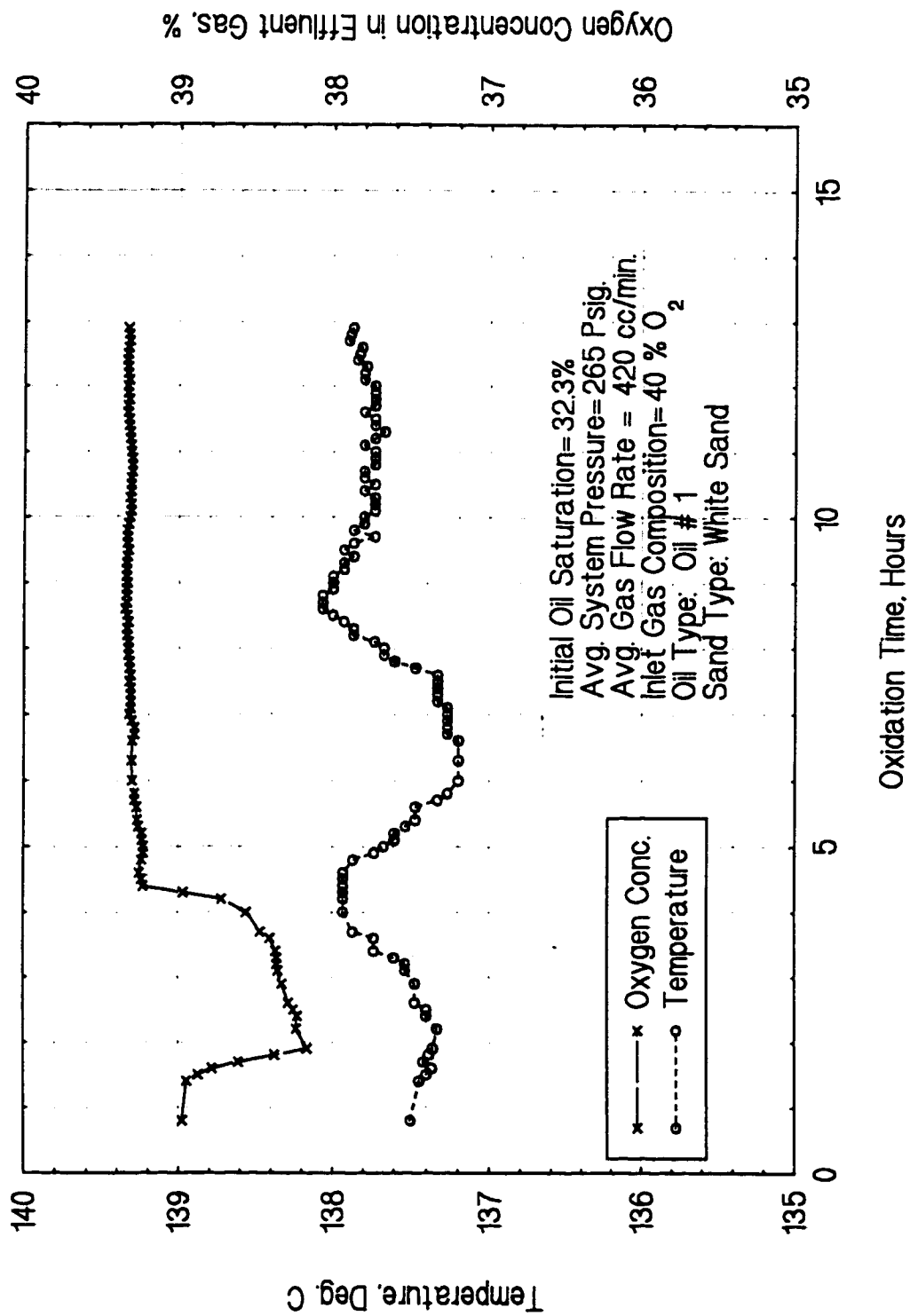


Figure 4.10: Low Temperature Oxidation Profile for Run # 06-SST

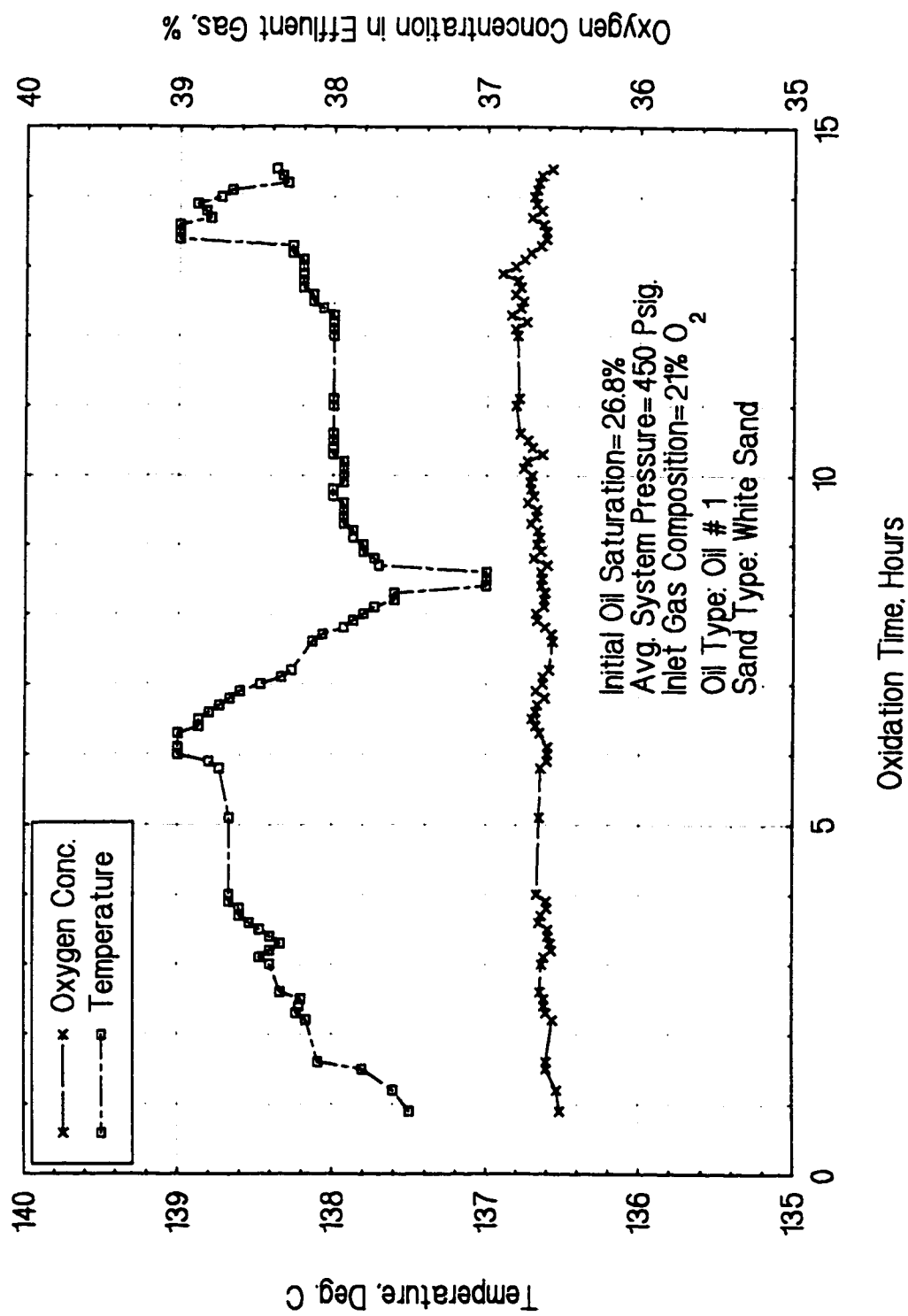


Figure 4.11: Low Temperature Oxidation Profile for Run # 07-SST

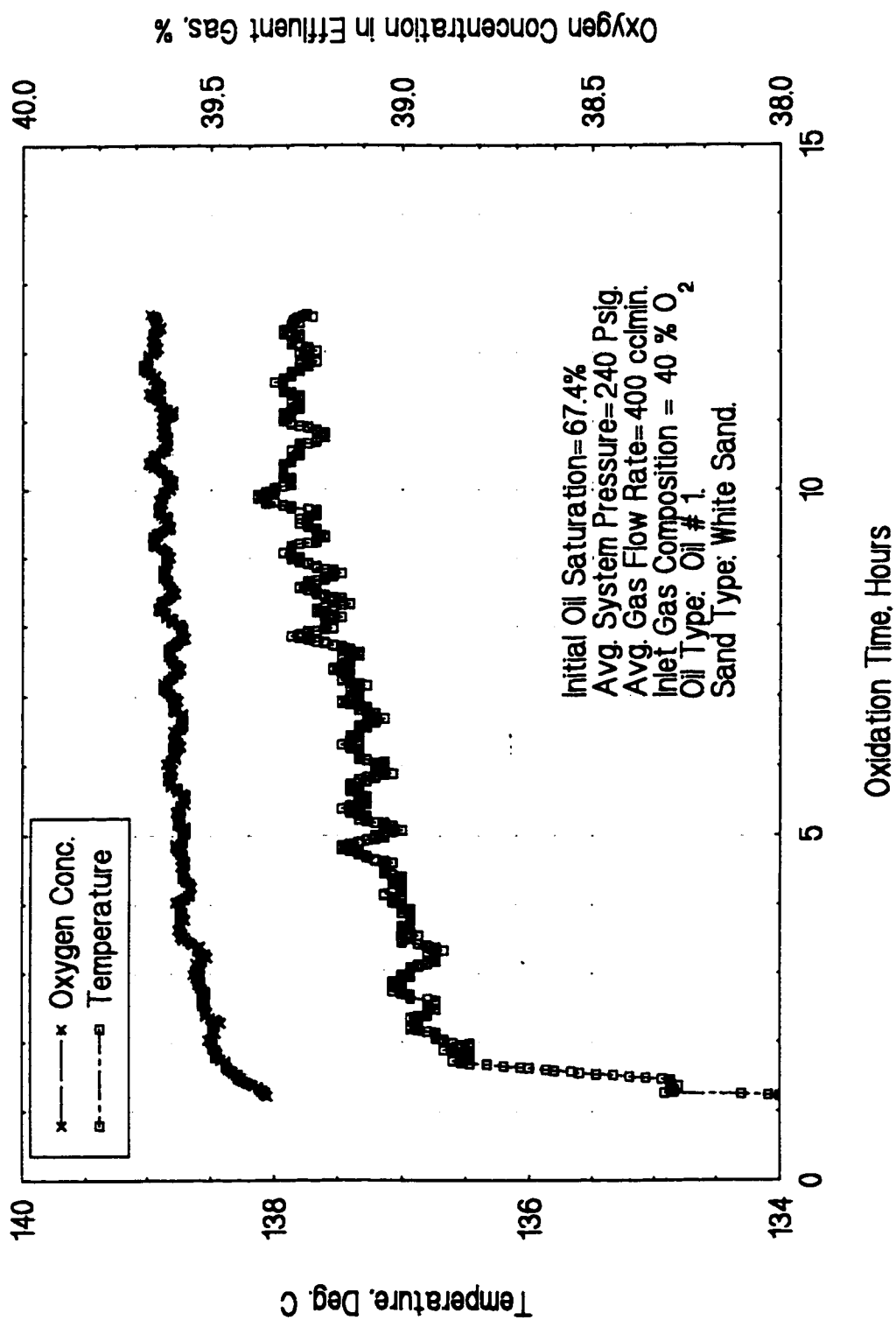


Figure 4.12: Low Temperature Oxidation Profile for Run # 08-SST

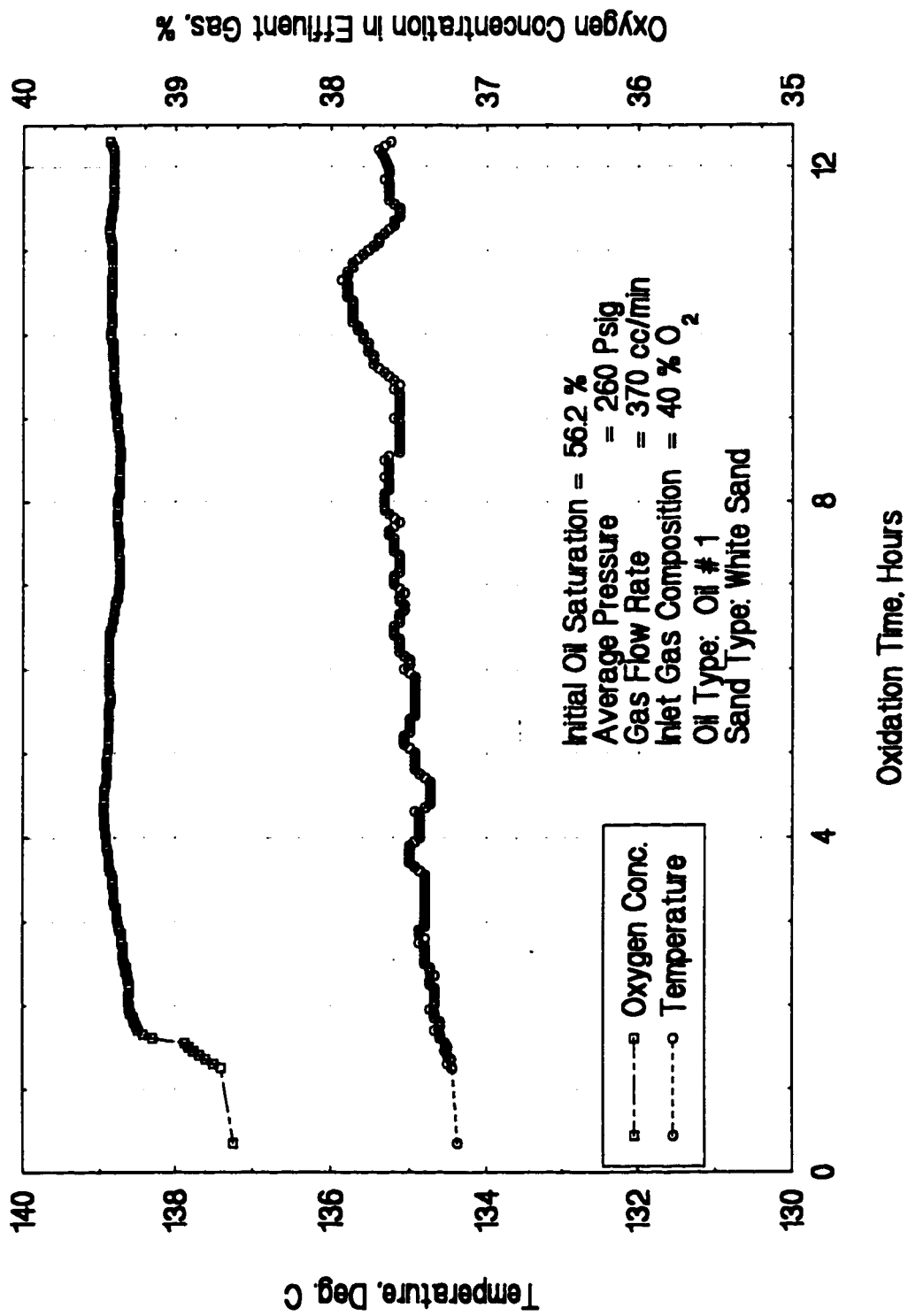


Figure 4.13: Low Temperature Oxidation Profile for Run # 01-BT

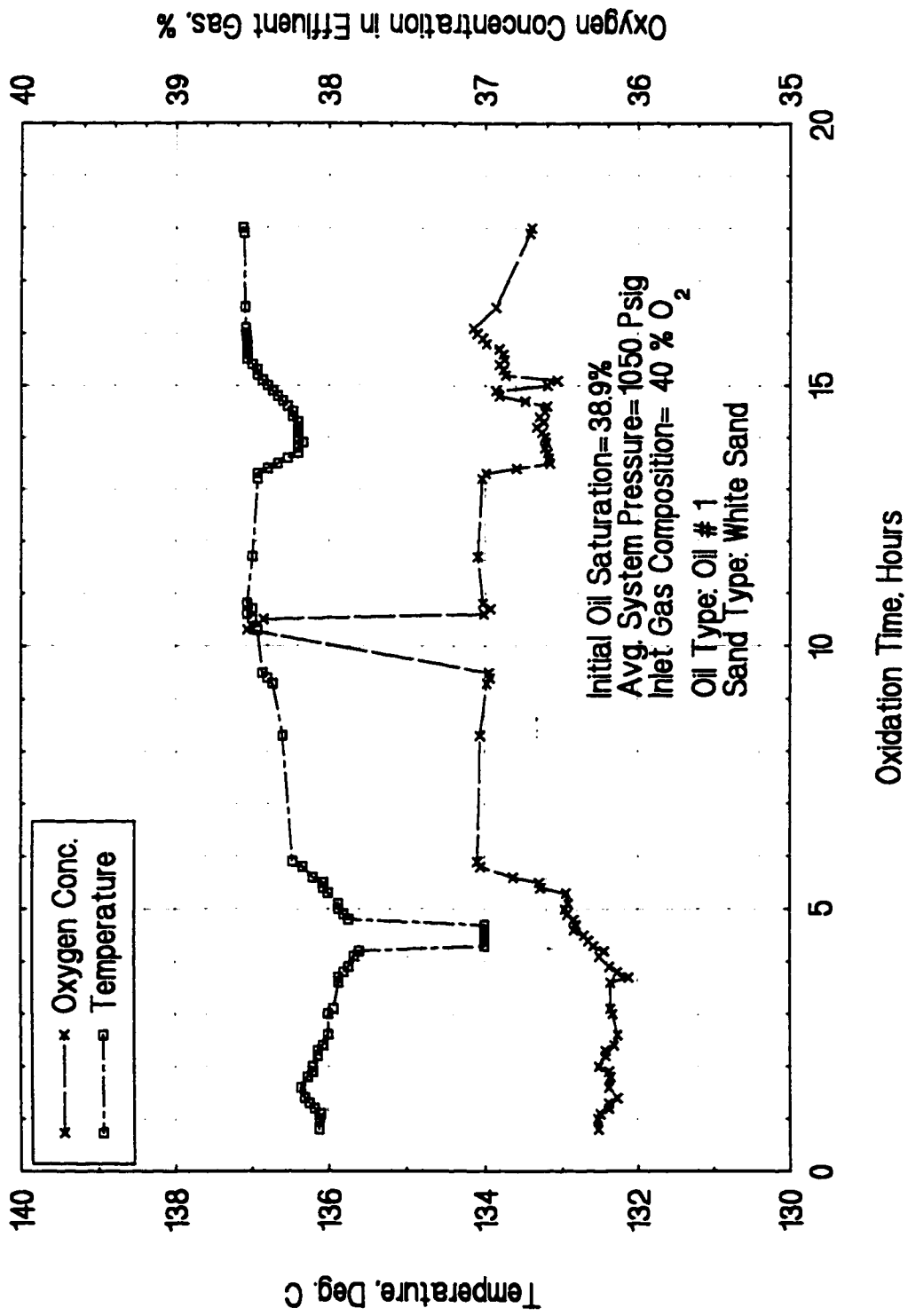


Figure 4.14: Low Temperature Oxidation Profile for Run # 02-BT

4.2.5 Oxidation Results using Reactor # 5

The most promising results yet obtained in this study are from the largest of all reactors, the 4.4-inch O.D Reactor # 5. As seen in Figure 4.16, within the first 5 hours of air injection, there was a temperature rise of 12 °C and oxygen concentration dropped by 2%. Thereafter, both parameters stayed constant, indicating extinction of LTO. This was at a gas flow rate of 250 cc/min. and 625 psig pressure using the enriched air. In the next run, seen in Figure 4.17, the temperature increased by 10 °C while oxygen concentration in the effluent gas dropped from 37% to 28.5%. These events occurred within a shorter period of 1.5 hours after commencement of air injection. Shortly after that, while the temperature was leveling off to a constant value, the oxygen concentration in the effluent began rising again towards the initial value of 40%, signaling extinction of the process.

The last run, seen in Figure 4.18, had the highest rise in temperature and largest drop in oxygen concentration of 14 °C and 34%, respectively, in just about ninety minutes after start of air injection. Shortly after this, however, the temperature started to drop and the oxygen concentration began to rise. This again indicated that there was no further heat build up, but rather cooling of the sand pack. This run was short-lived and abandoned prematurely after only 3 hours of air injection due to technical reasons.

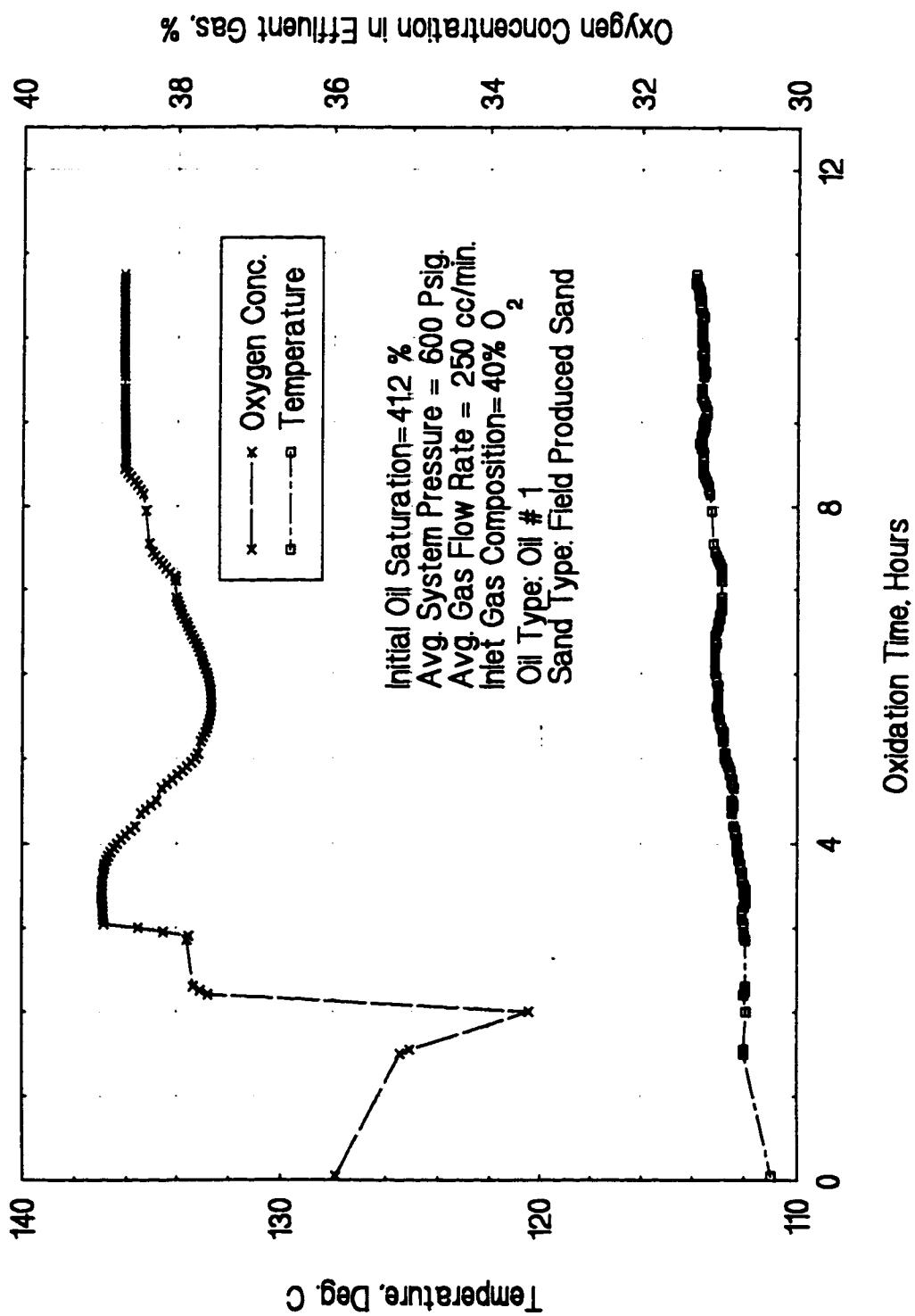


Figure 4.15: Low Temperature Oxidation Profile for Run # 01-LT

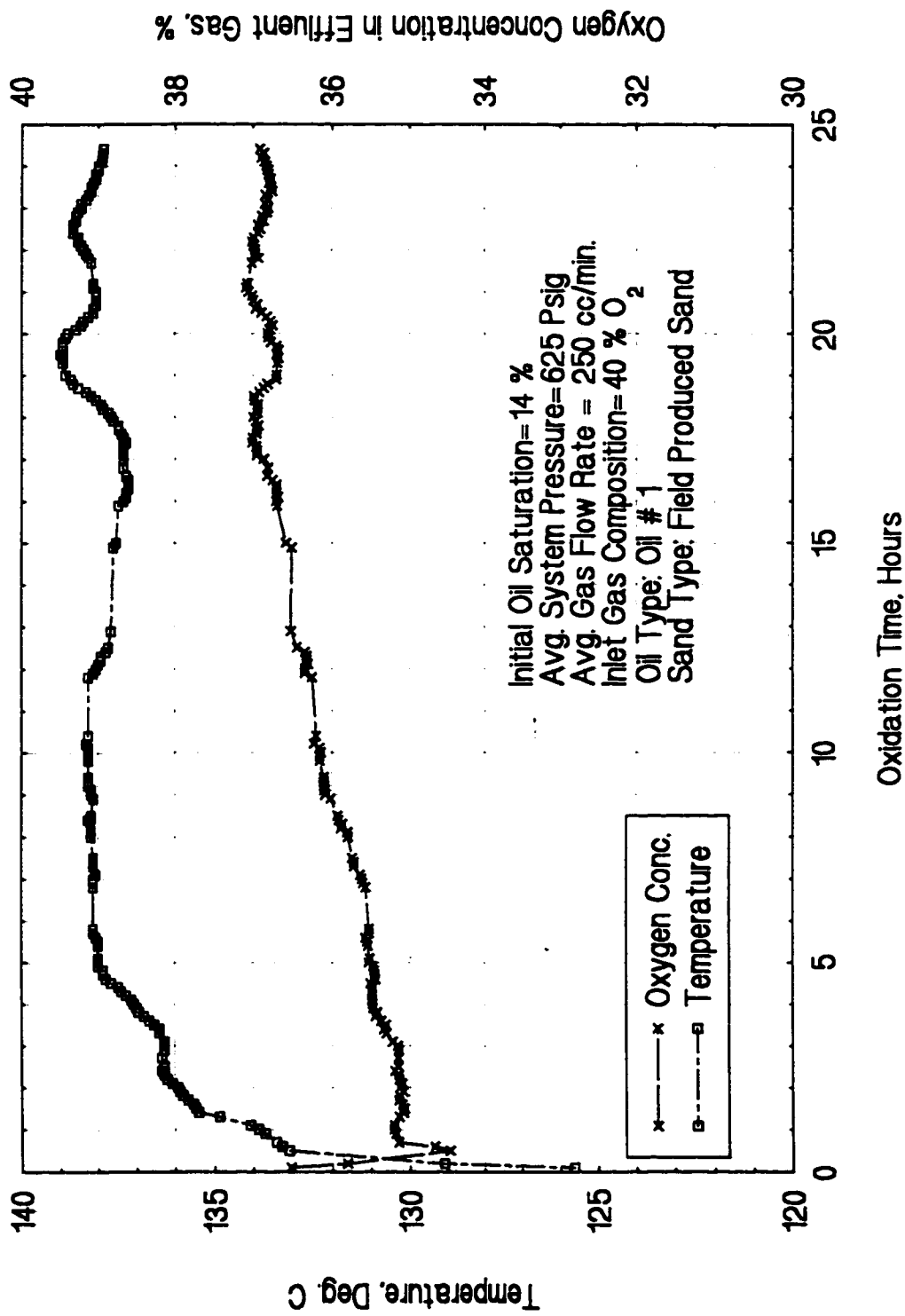


Figure 4.16: Low Temperature Oxidation Profile for Run # 02-LT

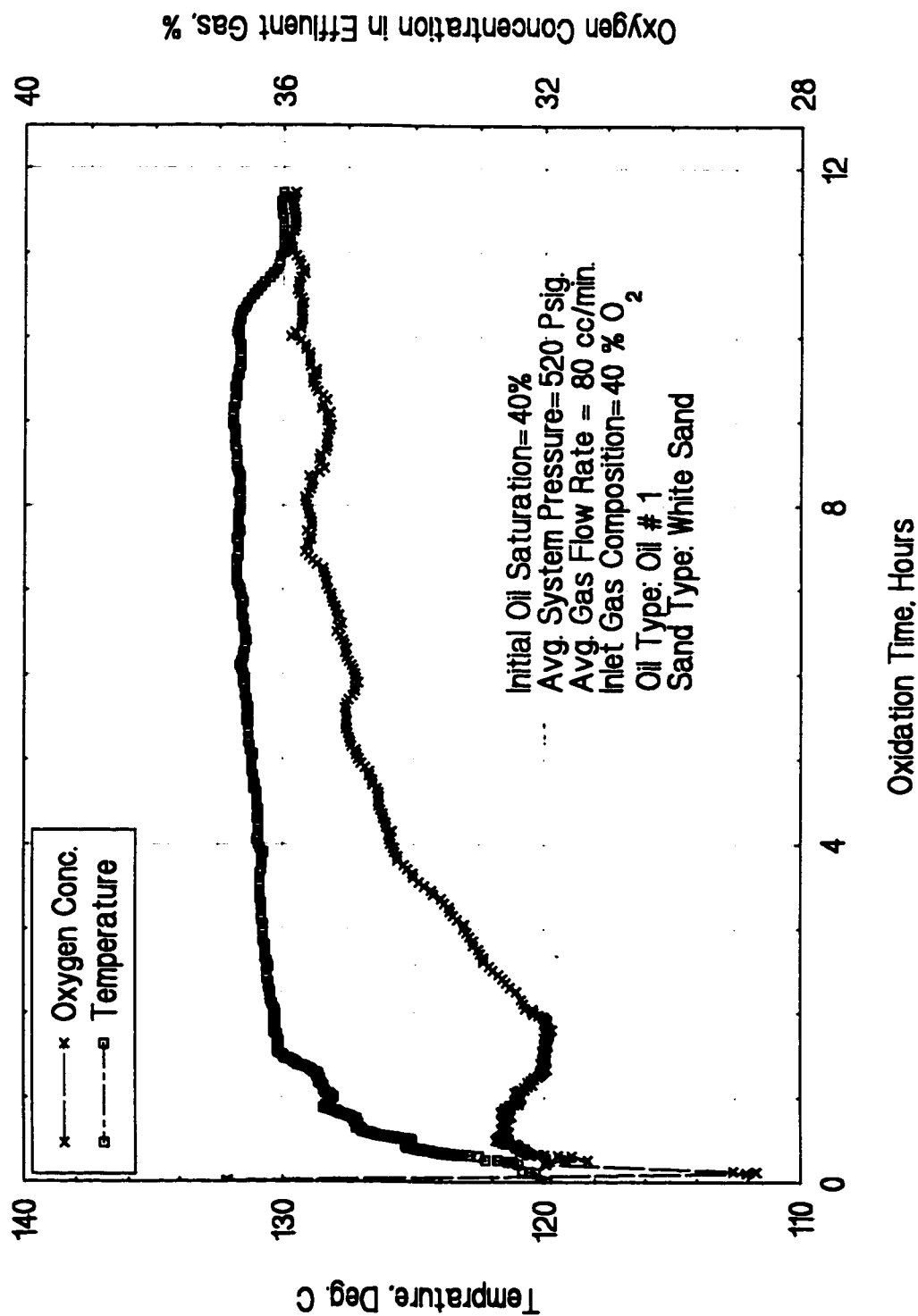


Figure 4.17: Low Temperature Oxidation Profile for Run # 03-LT

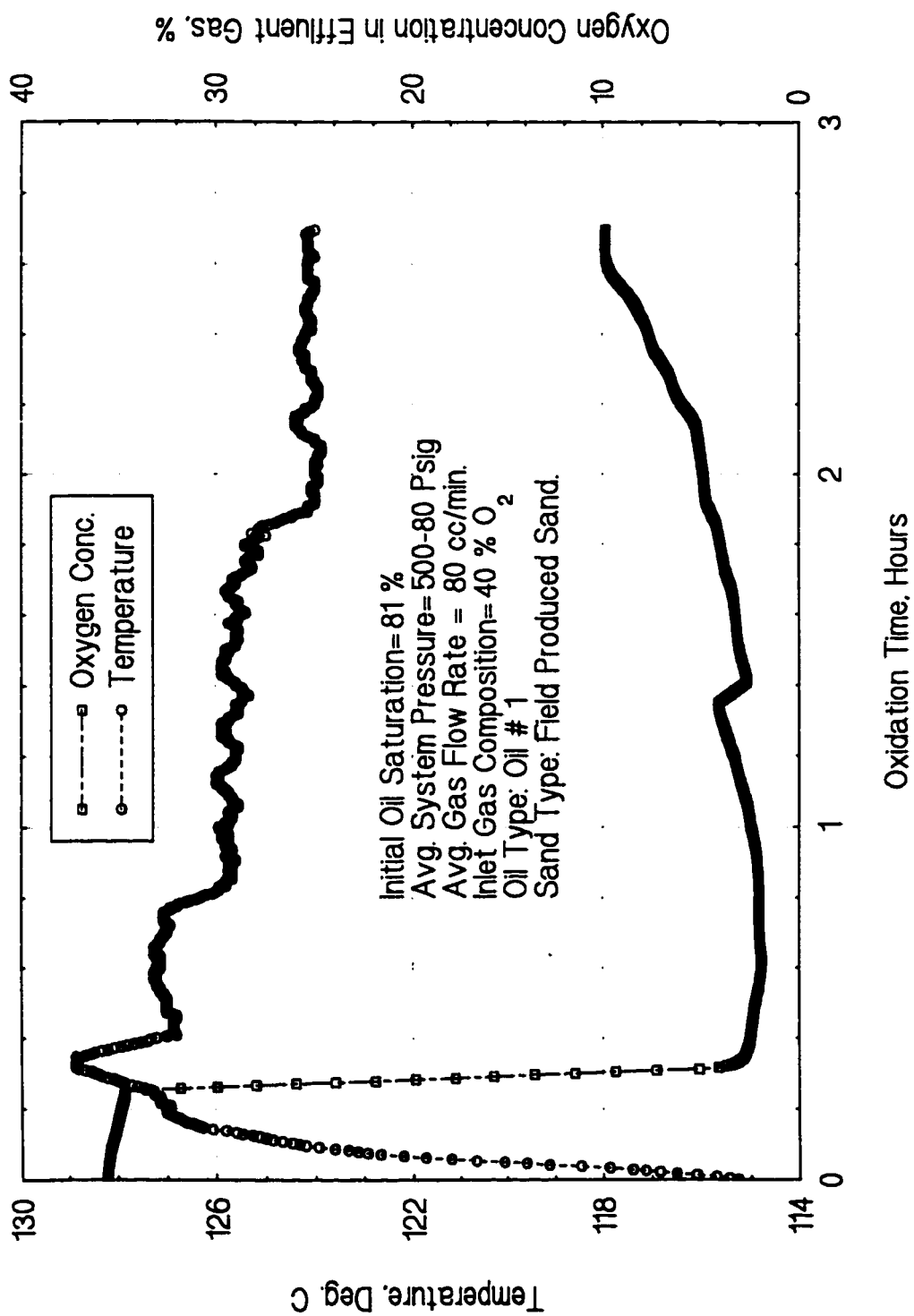


Figure 4.18: Low Temperature Oxidation Profile for Run # 04-LT

4.3 SUMMARY OF RESULTS OF OXIDATION RUNS

In an attempt to trigger spontaneous ignition by oxidation of the super-light-oil-saturated sand, successively larger reactors were used to increase the mass of the porous medium and enhance adiabatic conditions. Also, the initial oil saturation, the oxygen content of the oxidant and the operating pressure were increased. Almost all the runs, however, did not show any indications of spontaneous ignition, even after continued oxidation from 8-26 hours. This surprising observation was, however, quite contrary to the initial assumption that being an extremely light oil, Oil # 1 must be very reactive and should ignite spontaneously with air injection. In view of these results, it was concluded that Oil # 1, the super-light crude oil, is not susceptible enough to LTO to cause it to ignite spontaneously. The reason for this is attributable to the inherently low asphaltene content of the oil. As a result, it had become necessary to go back and examine the basic oxidation characteristics of the oil with the hope of coming up with an explanation for the poor ignition tendency of the oil. To achieve this, thermal analysis techniques were employed. A brief description of the process together with the experimental results are described next.

CHAPTER 5

EXPERIMENTAL WORK USING THERMAL ANALYSIS

5.1 PREVIEW OF THERMAL ANALYSIS METHODS.

Thermal analysis is an important approach when investigating the effect of temperature on the reactivity and/or chemical stability of a substance. Two commonly used techniques in thermal analysis are Thermogravimetric Analysis (TGA) and Differential Thermal Analysis (DTA). These are briefly described below.

(a) Thermogravimetric Analysis (TGA):

In TGA, a small sample of a substance is weighed continuously as it is heated at a constant rate. The resulting mass change versus temperature or time curve, also called the thermogram, provides valuable information regarding the thermal stability and composition of the initial sample, the thermal stability and composition of any intermediate compounds that may be formed and the composition of the residue, if any.

The derivative thermogravimetry, DTG, is the first derivative of the mass change curve. It consists of a series of peaks which are usually proportional to the total mass change of the sample, and by convention, is usually plotted on the same graph as the TG curve. It serves as a complimentary piece of information which makes it easier to analyze for small changes from one sample to the other[21]. Also, a DTG curve with its pronounced maxima clearly distinguishes between two changes that are close to each other in temperature.

(b) Differential Thermal Analysis (DTA):

DTA, on the other hand, is a thermal technique whereby the temperature of the sample compared with the temperature of a thermally inert substance is recorded as a function of the temperature of the sample, inert material or system, as the sample is heated or cooled at a uniform rate. The temperature changes occurring in the sample are due to chemical and physical changes such as endothermic, exothermic, enthalpic transition, oxidation and reduction reactions[20]. Characteristic features of a DTA curve include a steady initial rise indicating the onset temperature of the reaction or phase transition; an upward positive deflection will usually indicate an exothermic reaction while a downward negative deflection will indicate an endothermic process. The exothermic peak temperature indicates the maximum rate of heat evolution while the area under the curve is proportional to the heat of reaction[13].

Thermal analysis techniques have been used extensively in studying the thermal characteristics of crude oil oxidation and ignition. Tadema [16] in 1959 appears to be the first investigator who applied TDA to crude oil oxidation studies. By oxidizing oil/sand mixtures using air and nitrogen purging gases under the same conditions, he recognized two exothermic reactions: the first was LTO at 270 °C that resulted in the formation of a coke-like residue, with water as the major product of the reaction. The second occurred at 400 °C that produced mostly carbon oxides and very little water, and without any residue left behind. This reaction is now recognized as high temperature oxidation (HTO). This appeared to be the first initial recognition of these reaction regimes in oil oxidation studies.

In 1977, Bae[2] used both TG and DTA to study the thermo-oxidative behavior of fifteen crude oil samples with API gravities ranging from 6-38 degrees. He discovered that the thermo-oxidative behavior of the crude oils showed three distinct patterns, with certain distinguishing features that are directly dependant on the rapidity with which they are oxidized, thus providing a screening mechanism for crude oils for future thermal studies. In spite of these significant observations, Bae also found that the API gravities of these crude oil did not correlate well with the three patterns observed. In addition, he observed that the heat generated by LTO may be significant in fire flooding.

Carel and Cabiness[6] in 1981 reported a TGA technique to determine the oxidation and auto-ignition (spontaneous ignition)

characteristics of oil on lagging materials. The temperatures during the initial oxidation of the oil samples was easily observed from both the weight loss and the derivative curves, while the spontaneous ignition was determined from an acoustic source (auto-ignition sound).

The application of thermal analysis in this investigation is presented below.

5.2 The Experimental Setup

The main equipment for this investigation was the Simultaneous Thermal Analyzer, model STA-429, shown in Figure 5.1. It consists basically of three main components: the Measuring Chamber, the Recording and Controlling Cabinet and the Data Acquisition System. The Measuring Chamber houses the sample and reference crucibles. The sample to be measured is put in the sample crucible while the reference crucible contains Gamma Alumina powder of an identical weight to the sample. Gamma Alumina is inert and maintains the same weight throughout the heating process. Above both crucibles are their corresponding heating furnaces. The crucibles are attached to ultra sensitive thermobalances that monitor their weights as heating is progressing. The oxidant for the experiment is supplied by compressed gas cylinders connected to a flow meter on the Measuring Chamber by plastic tubings.

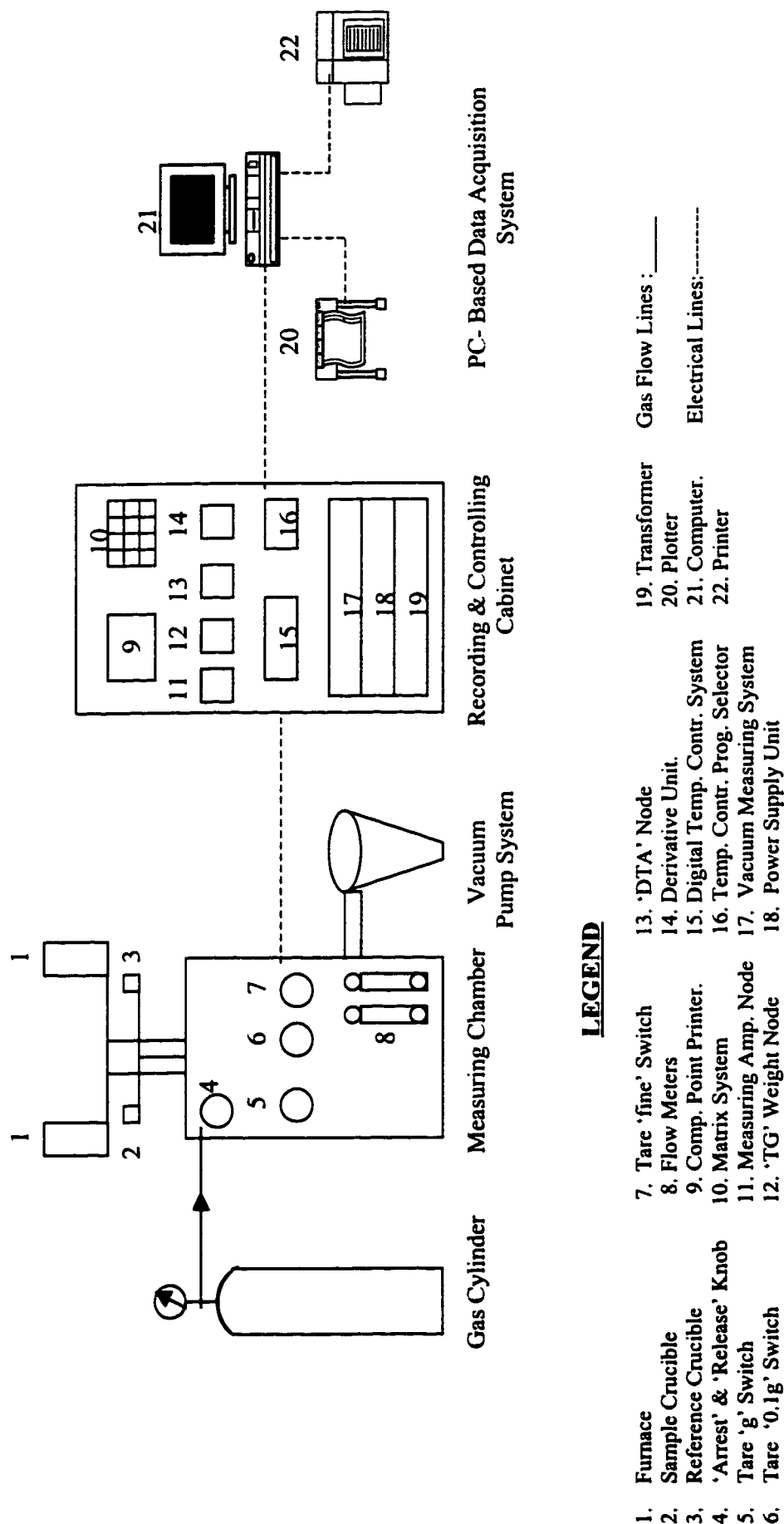


Figure 5.1: Thermogravimetric Analysis Equipment and Set-Up

5.3 Experimental Procedure

An experimental run is usually started by placing about a 100 milligram of the sand/oil mixture in the sample crucible. The furnaces are then lowered over both crucibles and secured in place. The oxidant gas is turned on and the flow meter adjusted to give a constant flow rate of 100 cubic centimeters per minute. Heating is then commenced at the Recording and Controlling Cabinet while data acquisition is begun simultaneously. A run will normally take about an hour and a half. The experimental conditions maintained in all runs are as listed below.

| | | |
|--------------------|---|----------------|
| Sample Weight | = | 100 milligrams |
| Heating Rate | = | 10 °C/min. |
| Gas Injection Rate | = | 100 cc/min. |
| System Pressure | = | Atmospheric |

5.4 RESULTS AND DISCUSSION OF THERMAL ANALYSIS

Figure 5.2 shows the thermogram for the first test, which was conducted on a mixture of Oil # 1 and the produced sand. The initial oil saturation in the sample was 40 %. Like most thermograms, the figure consists of two ordinates and one abscissa. The left ordinate represents the mass loss of the sample in percent while the right ordinate shows the differential temperature in micro-volts. The abscissa is the temperature

in degrees Celsius. Three curves are shown on the graph, the TG, DTG and DTA. The TG shows that there was a mass loss of 2.8% from ambient to 190 °C, a sudden mass loss of 3.55% between 190 – 320 °C and a very slow mass loss of 1.14% from 320 – 550 °C. Thereafter, there was no significant mass loss as the curve levels off. These mass changes are shown in Table 5.1. The DTG which gives a more accurate indication of thermal changes during the oxidation process shows that LTO begins at 170 °C, indicated by the end of first depression of the curve, and reaches a maximum at 262 °C, seen as the lowest point of the second depression. At the LTO peak temperature of 262 °C, the corresponding DTA value is 55 micro-volts, as seen on Figure 5.2. This differential temperature is the difference between the temperature of the reference sample and that of the sample at the time of LTO. This is related to how much heat is released at LTO, and it gives an indication of how susceptible a crude oil is to LTO which is, ultimately, a measure of the potential of the crude oil to spontaneous ignition. Spontaneous ignition will usually be indicated by a very high differential temperature.

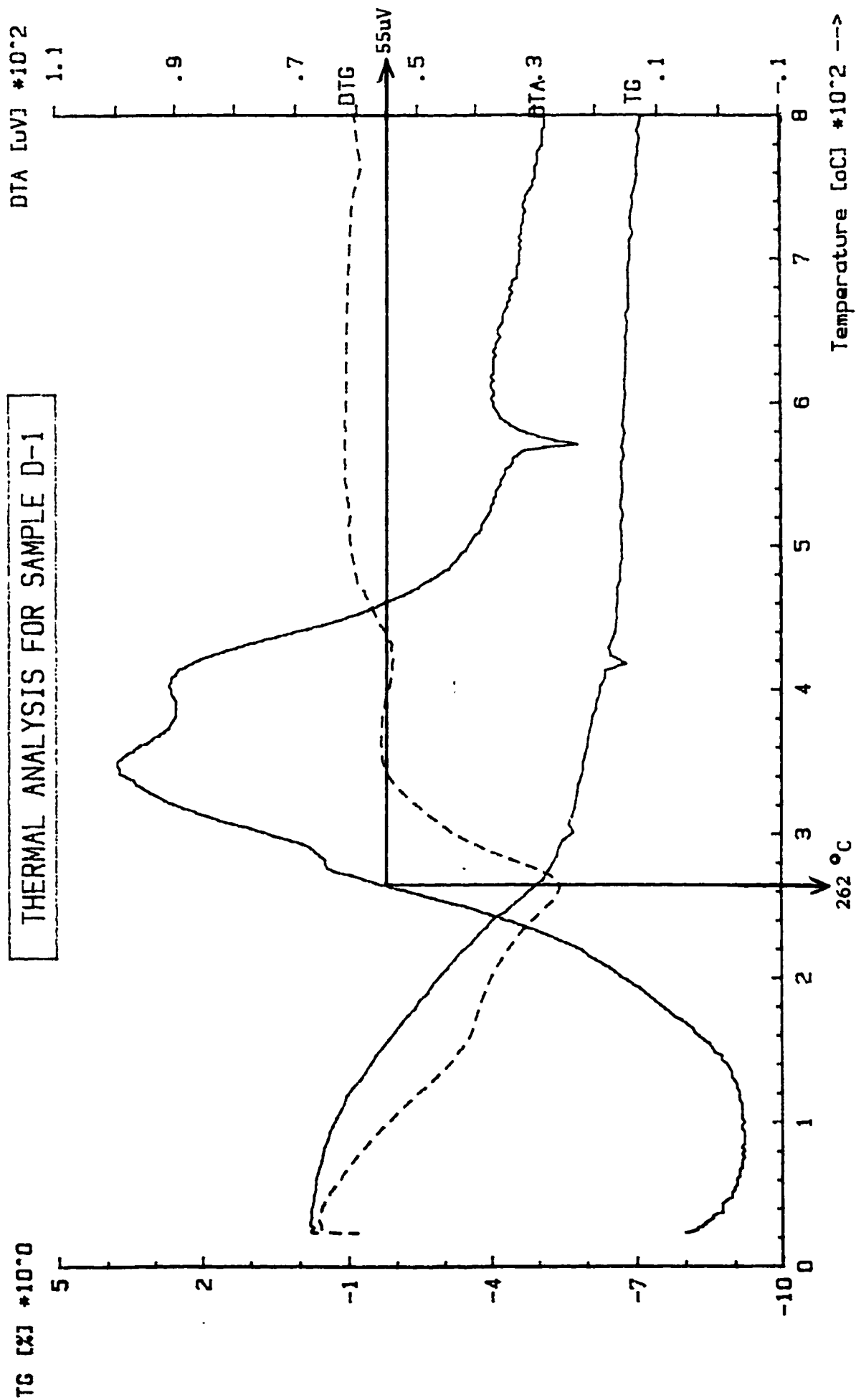


Figure 5.2: Thermal Analysis Curves for Oil # 3.

Table 5.1: Thermogravimetric Mass Loss for Oil # 1

| CRUDE OIL | TEMPERATURE RANGE (°C) | THERMAL EFFECT | T-max OF DTA PEAK (°C) | MASS LOSS (%) | TOTAL MASS LOSS (%) |
|-----------------|------------------------|----------------|------------------------|---------------|---------------------|
| Oil Sample # 1. | 20 – 190 | Endothermic | 180 | 2.81 | 7.5 |
| | 190-350 | Exothermic | 280 | 3.55 | |
| | 350-550 | Exothermic | 410 | 1.14 | |

In the following paragraphs, the effects of various process parameters on the thermogravimetric behavior of the oil/sand systems are discussed.

5.4.1 Effects of Crude Oil Type:

Oils # 1, 2 and 3 mixed with the produced sand were subjected to thermal analysis under the same experimental conditions. The TG, DTG and DTA curves resulting from these runs are shown in Figures 5.3 and 5.4. From these curves it can be observed that all three oils demonstrated the same LTO peak temperature of 262 °C. However, at this LTO peak, the differential temperature for Oils # 2 and 3 were 110 and 90 micro-volts, respectively, which were higher than that of Oil # 1, which was 55 micro-volts. This shows that Oil # 2 releases about twice as much heat as the super-light Oil # 1, and therefore more reactive at the same LTO temperature. It should be remembered that Oil # 2 has a higher asphaltene content (4.1%) than that of Oil # 1 (0.18%). The corresponding mass losses and thermal effects are listed in Table 5.2.

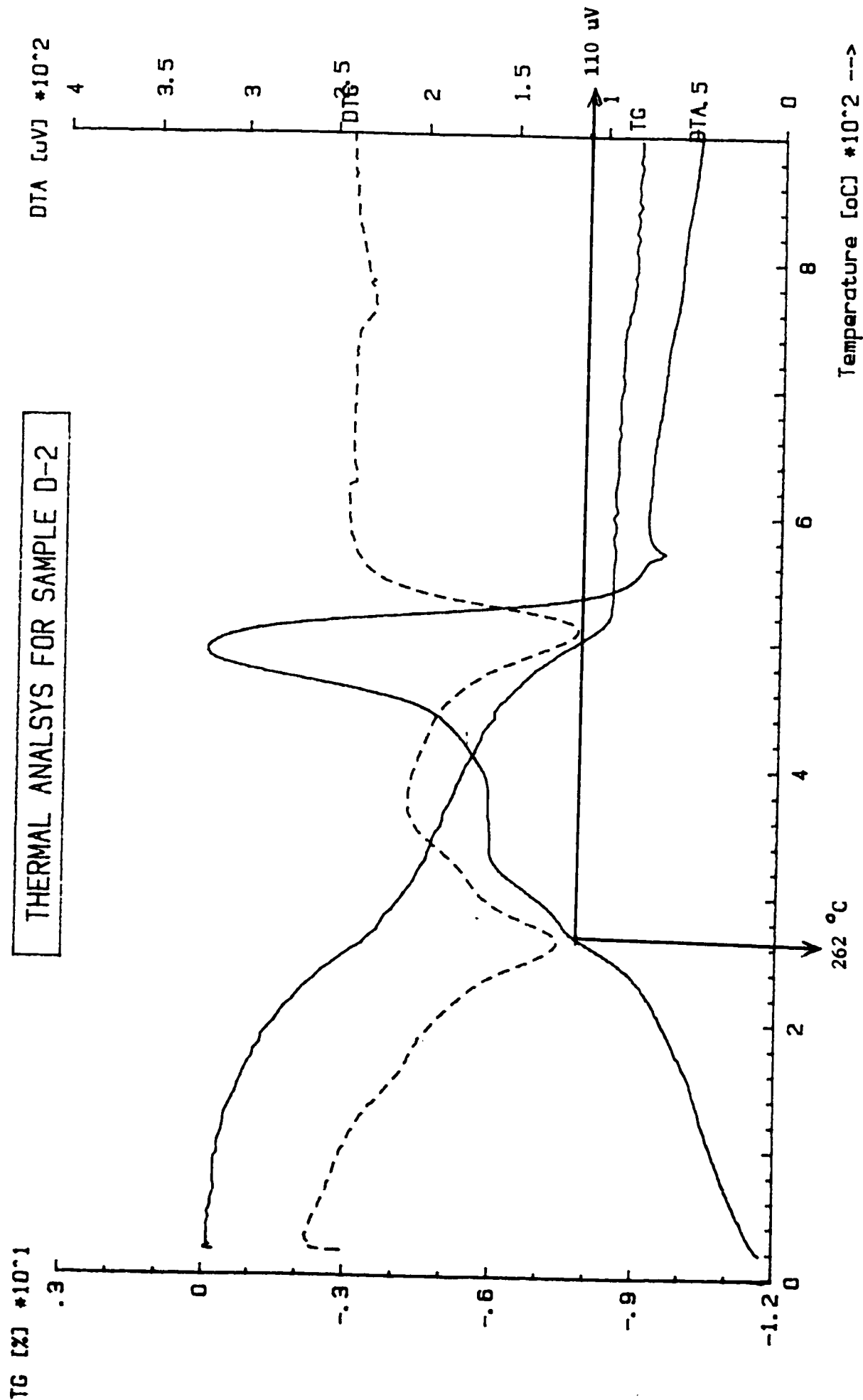


Figure 5.3: Thermal Analysis Curves for Oil # 2.

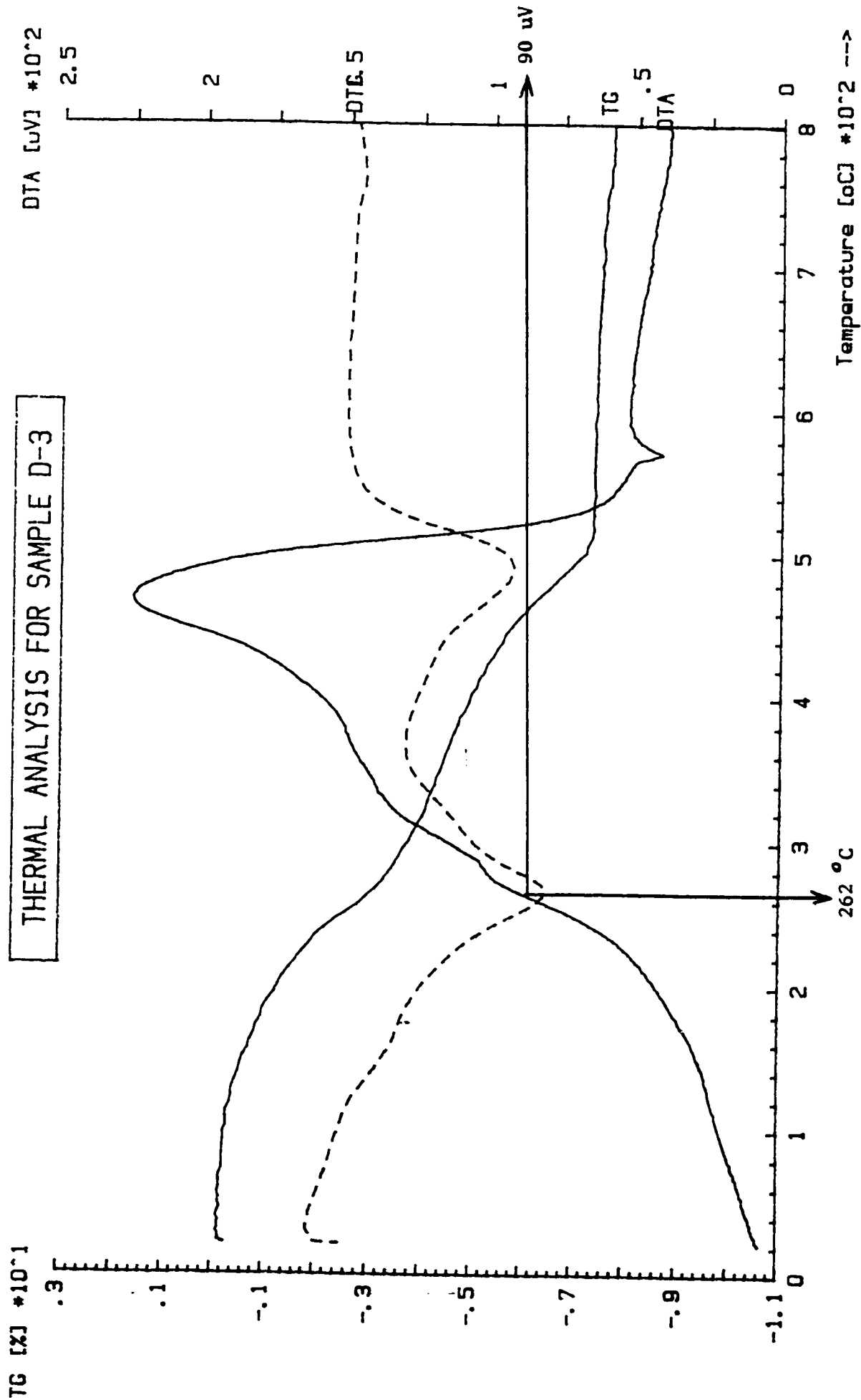


Figure 5.4: Thermal Analysis Curves for Oil # 3.

Table 5.2: TG Mass Loss for the Different Crude Oil Types

| CRUDE OIL IN MIXTURE | TEMPERATURE RANGE (°C) | THERMAL EFFECT | T-max DTA PEAK (°C) | MASS LOSS (%) | TOTAL MASS LOSS (%) |
|---------------------------------|---------------------------------------|---------------------------|------------------------------------|--------------------------|------------------------------------|
| Oil # 1 | 20 – 190 | Endothermic | 180 | 2.81 | 7.5 |
| | 190 – 320 | Exothermic | 280 | 3.55 | |
| | 320 – 550 | Exothermic | 410 | 1.14 | |
| Oil # 2 | 20 – 190 | Endothermic | 180 | 1.33 | 8.30 |
| | 190 – 320 | Exothermic | 280 | 3.34 | |
| | 320 – 550 | Exothermic | 410 | 3.63 | |
| Oil # 3 | 20 – 190 | Exothermic | 180 | 0.88 | 7.2 |
| | 190 – 320 | Exothermic | 280 | 2.96 | |
| | 320 – 550 | Exothermic | 410 | 3.36 | |

5.4.2 Effects of Oxygen Partial Pressures

By comparing the thermograms of Oil # 1 oxidized in air (Figure 5.2) with the one oxidized in the enriched air of 40% oxygen concentration (Figure 5.5), it is found that at the LTO peak the sample oxidized in the enriched air had a much higher differential temperature (145 micro-volts) than that oxidized in ordinary air (55 micro-volts). As expected, the oxidant with the higher oxygen concentration enhances a faster rate of oxidation and will induce spontaneous ignition faster and more effectively. Table 5.3 lists the corresponding thermogravimetric mass losses.

5.4.3 Effects of Initial Water Saturation

The thermogram in Figure 5.6 was obtained by oxidizing oil #1/sand mixture in which both the initial oil and water saturations were 40%. This was compared to the thermogram of the usual mixture which had only 40% initial oil saturation (figure 5.2), for the purpose of examining the effect of the presence of water. As can be seen from the two thermograms, the mixture with an initial water saturation had a differential temperature of 88 micro-volts whereas the one without water had 55 micro-volts, thus more heat was released in the presence of water than in its absence. However, the difference does not seem to be very high. It can therefore be inferred from the above that presence of water does not significantly affect the rate of oxidation.

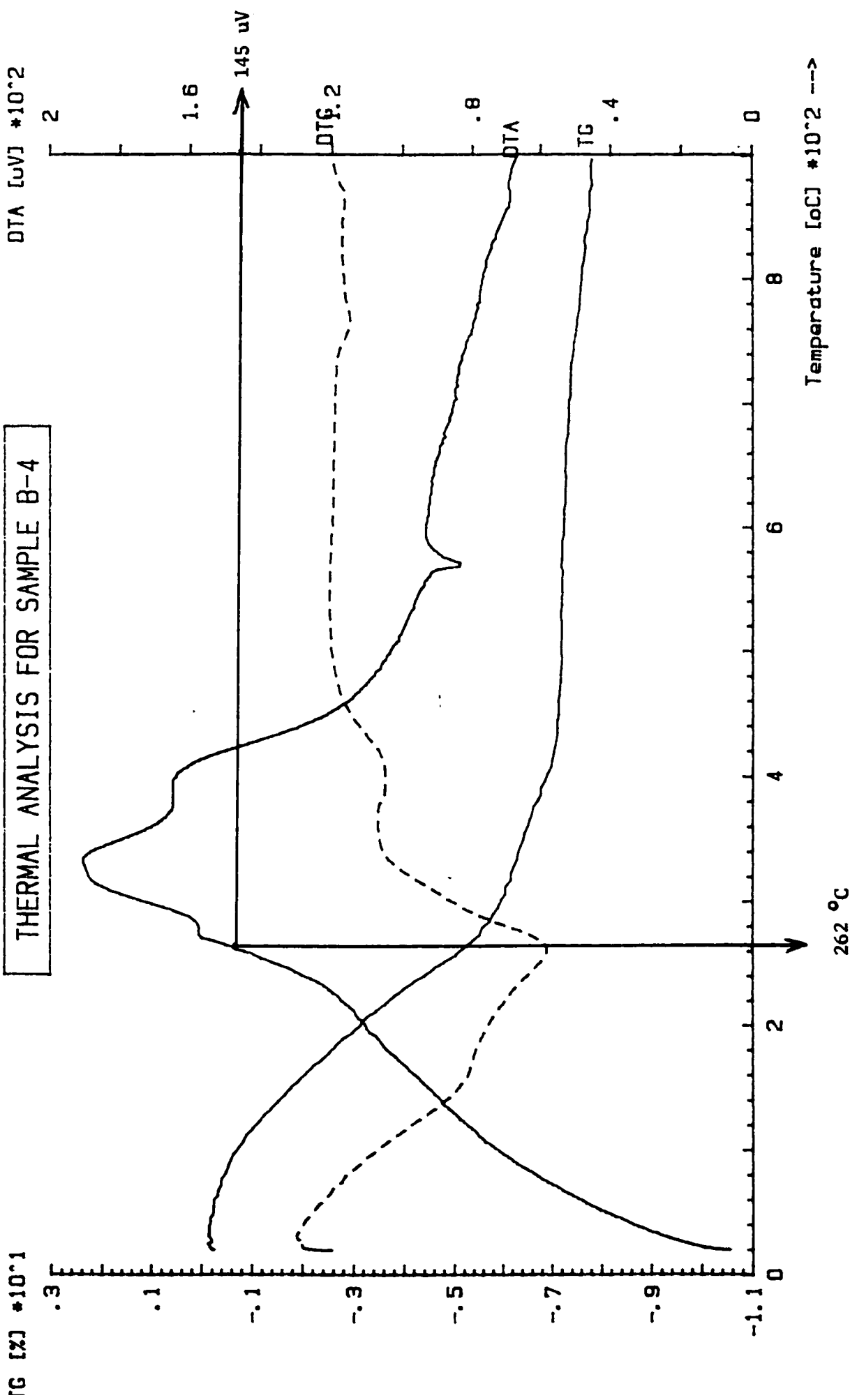


Figure 5.5: Thermal Analysis Curve for Oil # 1 Oxidized in Mixed Gas (40% O₂).

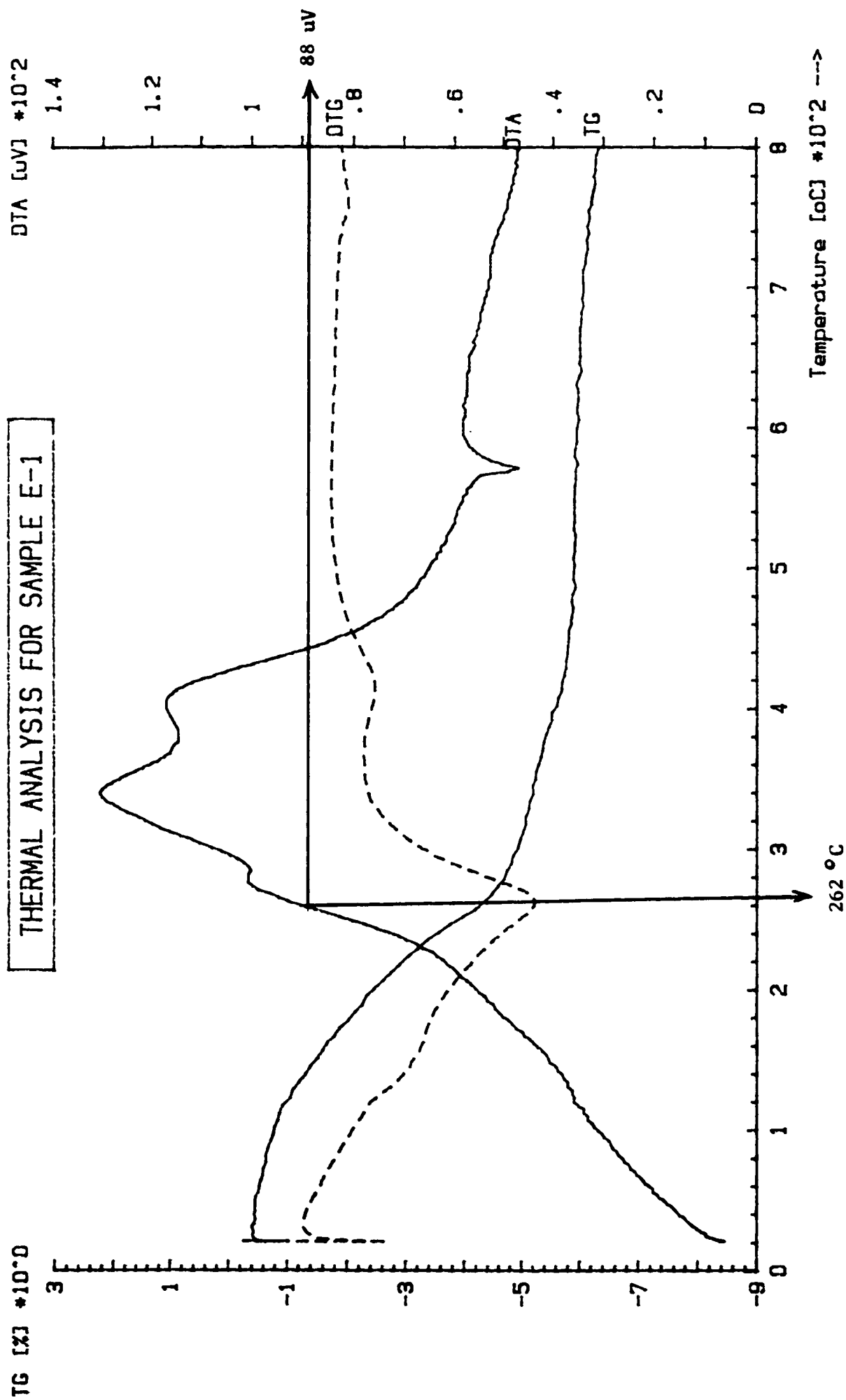


Figure 5.6: Thermal Analysis Curve for Oil # 1 Oxidized with Initial Water Saturation.

Table 5.3: Thermogravimetric Mass Loss of Oil # 1, for Different Oxygen Partial Pressures

| PERCENT OXYGEN IN PURGING GAS (%) | TEMPERATURE RANGE °C | THERMAL EFFECT | T-max OF DTA PEAK °C | MASS LOSS (%) | TOTAL MASS LOSS (%) |
|--|-------------------------------------|---------------------------|---|--------------------------|------------------------------------|
| 21 | 20-190 | Endothermic | 180 | 2.81 | 7.5 |
| | 190-320 | Exothermic | 280 | 3.55 | |
| | 320-550 | Exothermic | 410 | 1.14 | |
| 40 | 20-190 | Exothermic | 180 | 2.61 | 7.02 |
| | 190-320 | Exothermic | 280 | 3.15 | |
| | 320-550 | Exothermic | 410 | 1.26 | |

5.5 SUMMARY OF THE OBSERVED EFFECTS OF PROCESS PARAMETERS ON TG/DTA CURVES

- (i) All crude oils have the same temperature at which LTO peaks.
- (ii) Crude oils with a larger asphaltene content tend to release more heat over the LTO regime.
- (iii) Oxidants of higher oxygen concentrations tend to accelerate oxidation more than those with less oxygen concentrations
- (iv) The presence of initial water saturation did not significantly affect TG/DTA curves, neither did it affect the LTO and HTO onset and peak temperatures.

CHAPTER 6

CONCLUSIONS

Based on the results of this study, the following conclusions can be made:

- (1) Under the various experimental conditions of this investigation, spontaneous ignition has not been achieved on the super-light crude, Oil # 1.
- (2) Even though the different crude oils showed the same LTO peak temperatures, as evidenced from thermal analysis, the heat generated by the super-light oil was however less than the heavier crudes
- (3) Inability of the super-light crude oil to self ignite is then attributable to insufficient heat generation during LTO.
- (4) Small amount of heat released during LTO of the super-light oil is probably due to its low asphaltene content.

- (5) Therefore, air injection into the super-light oil reservoir will, in most probability, not pose a risk of spontaneous ignition. However, whether this oil, with its lack of asphaltenes, can be a proper sand consolidation medium is subject to investigation.

REFERENCES

- (1) Alexander, J.D., Martin, W.L. and Dew, J.N.: "Factors Affecting Fuel Availability and Composition During In-Situ Combustion," JPT, October, 1962, pp. 1154-1162
- (2) Bae, J.H.: "Characterization of Crude Oil for Fireflooding Using Thermal Analysis Methods," SPEJ, June, 1977, pp. 211-218
- (3) Bousaid, I.S. and Ramey, H.J., Jr.: "Oxidation of Oil in Porous Media," SPEJ, June, 1968, pp. 137-148
- (4) Burger, J.G.: "Spontaneous Ignition in Oil Reservoirs," SPEJ, April, 1976, pp. 73-81.
- (5) Burger, J.G. and Sahuquet, B.C.: "Chemical Aspects of In-Situ Combustion - Heat of Combustion and Kinetics," SPEJ, October, 1972. pp. 410-422.
- (6) Carel, A.B. and Cabiness, D.K.: "Thermal Method for Determining the Oxidation-Autoignition Characteristics of Oils," Thermochemica Acta, 44 (1981), pp. 363-367.

- (7) Caruthers, R.M.: "High Temperature Oxidation of Crude Oil in Porous Media," SPE Paper # 2913, 1965.
- (8) Caruthers, R.M.: "Spontaneous Ignition in Porous Media," PhD Dissertation, University of Texas, 1965.
- (9) Dabbous, M.K. and Fulton, P.F.: "Low-Temperature Oxidation Reaction Kinetics and Effects on the In-Situ Combustion Process," SPEJ, June, 1974, pp. 253-262.
- (10) Fassihi, M.R, Brigham, W.E and Ramey, H.J. Jr.: "Reaction Kinetics of In-Situ Combustion, Part 1," Observations. SPEJ, August, 1984, pp. 399-407.
- (11) Fassihi, M.R., Meyers, K.O. and Basslie, P.F.: "Low-Temperature Oxidation of Viscous Crude Oils," SPEJ, November, 1990, pp. 609-616
- (12) Howard, C.E., Widmeyer, R.H. and Haynes, S. Jr.: "The Charco Redondo Thermal Recovery Pilot," J. Pet. Tech. Dec. 1977, pp. 1522-1532.
- (13) Pope, M.I., and Judd, P.D.: "Differential Thermal Analysis." Heyden and Sons Publications, London, U.K., 1997. Pp30-31.
- (14) Prats, M.: "Thermal Recovery," SPE Monograph Series, Vol. 7, 1982, pp. 88-110.

(15) Strange, L.K.: "Ignition, Key Phase In Combustion Recovery," Petroleum Engineer, November 1964. pp. 105-109.

(16) Tadema, H.J.: "Mechanism of Oil Production by Underground Combustion," Proc, Fifth World Petroleum Congress, New York, 1959, pp. 279

(17) Tadema, H.J. and Quant, J. T.: "Process for Igniting Hydrocarbon Materials Present within Oil-Bearing Formations," U.S. Patent No. 2,863, 510, December 9, 1958, pp. 1-4.

(18) Tadema, H.J. and Weijdema, J.: "Spontaneous Ignition of Oil Sands," Oil and Gas J. December 14, 1970, pp. 77-80

(19) Warren, J.E., Reed, R. L., and Price, H.S.: "Theoretical Consideration of Reverse Combustion in Tar Sands," Trans AIME (1960) 219, 109.

(20) Wendlandt, W.W.M.: "Thermal Methods of Analysis," John Wiley and Sons Publications, New York City, 1974, pp. 3-136.

(21) Wayland, J.R., Lee, D.O. and Massis, T.M.: "On the application of TG Concepts to 'In-Situ' Combustion Processes," Sandia Report, June 1983, pp. 4-5.

A P P E N D I X

Table A-1

Experimental Results for Run# 02-ST

Initial Oxidation Temperature = 102 deg. C
Average System Pressure = 102 Psig.

Inlet Gas Composition = 21% O₂
Average Gas Flow Rate = 600 cc/min

| Time, Hrs. | Temp. °C | O ₂ Conc. % | Time, Hrs. | Temp. °C | O ₂ Conc. % |
|------------|----------|------------------------|------------|----------|------------------------|
| 1.50 | 137.83 | 14.82 | 9.50 | 137.00 | 21.15 |
| 1.60 | 138.14 | 12.71 | 9.60 | 136.91 | 21.20 |
| 1.70 | 138.50 | 11.13 | 9.70 | 136.73 | 21.20 |
| 1.80 | 138.78 | 9.89 | 9.80 | 136.91 | 21.17 |
| 1.90 | 139.00 | 8.91 | 9.90 | 137.00 | 21.13 |
| 2.00 | 139.09 | 8.10 | 10.00 | 137.00 | 21.10 |
| 2.10 | 139.64 | 3.59 | 10.10 | 137.00 | 21.07 |
| 2.20 | 140.18 | 3.98 | 11.50 | 137.82 | 20.84 |
| 2.30 | 140.55 | 4.58 | 11.60 | 137.82 | 20.84 |
| 2.40 | 140.82 | 7.55 | 11.70 | 137.91 | 20.82 |
| 2.50 | 139.36 | 9.47 | 11.80 | 138.09 | 20.80 |
| 2.70 | 139.00 | 11.34 | 11.90 | 138.09 | 20.78 |
| 2.80 | 138.64 | 13.19 | 12.00 | 138.27 | 20.76 |
| 2.90 | 138.27 | 15.07 | 12.10 | 138.27 | 20.75 |
| 3.00 | 138.00 | 16.95 | 12.20 | 138.36 | 20.73 |
| 0.33 | 125.28 | 18.82 | 12.30 | 138.36 | 20.71 |
| 6.20 | 137.36 | 20.68 | 12.40 | 138.36 | 20.70 |
| 6.30 | 137.18 | 20.83 | 12.50 | 138.27 | 20.70 |
| 6.40 | 136.82 | 20.75 | 12.60 | 138.27 | 20.70 |
| 6.50 | 136.36 | 20.66 | 12.70 | 138.36 | 20.70 |
| 6.60 | 135.82 | 20.59 | 12.80 | 138.36 | 20.70 |
| 6.70 | 137.00 | 20.52 | 12.90 | 138.27 | 20.70 |
| 6.80 | 137.18 | 20.51 | 13.00 | 138.27 | 20.70 |
| 6.90 | 137.18 | 20.51 | 13.10 | 138.09 | 20.70 |
| 7.00 | 137.27 | 20.48 | 13.20 | 138.00 | 20.70 |
| 7.10 | 137.36 | 20.50 | 13.30 | 138.00 | 20.70 |
| 7.20 | 137.55 | 20.52 | 13.40 | 137.91 | 20.70 |
| 7.30 | 137.55 | 20.54 | 13.50 | 137.82 | 20.70 |
| 7.40 | 137.46 | 20.55 | 13.60 | 137.82 | 20.70 |
| 7.50 | 137.46 | 20.57 | 13.70 | 137.82 | 20.70 |
| 8.60 | 137.55 | 20.65 | 13.80 | 137.82 | 20.70 |
| 8.70 | 137.55 | 20.72 | 13.90 | 137.91 | 20.70 |
| 8.80 | 137.55 | 20.79 | 14.00 | 138.00 | 20.70 |
| 8.90 | 137.46 | 20.85 | 14.10 | 137.91 | 20.73 |
| 9.00 | 137.55 | 20.92 | 14.20 | 138.00 | 20.73 |
| 9.10 | 137.36 | 20.99 | 14.30 | 138.11 | 20.73 |
| 9.20 | 137.18 | 21.02 | 14.40 | 138.13 | 20.74 |
| 9.30 | 137.18 | 21.05 | 14.50 | 138.14 | 20.74 |
| 9.40 | 137.00 | 21.10 | 15.10 | 138.33 | 20.75 |

Table A-2

Experimental Results for Run # 01-SST

Initial Oxidation Temperature = 136 deg. C
Average System Pressure = 400 Psig.

Inlet Gas Composition = 21 % O₂
Average Gas Flow Rate = 600 cc/min

| Time, Hrs. | Temp. °C | O ₂ Conc. % | Time, Hrs. | Temp. °C | O ₂ Conc. % |
|------------|----------|------------------------|------------|----------|------------------------|
| 1.40 | 142.00 | 20.80 | 5.60 | 143.00 | 20.90 |
| 1.50 | 142.00 | 20.80 | 5.70 | 143.00 | 20.80 |
| 1.60 | 142.00 | 20.90 | 5.80 | 143.00 | 20.90 |
| 1.70 | 142.00 | 20.80 | 6.00 | 143.00 | 20.80 |
| 1.80 | 141.00 | 20.80 | 6.20 | 142.00 | 20.80 |
| 1.90 | 143.00 | 20.70 | 6.30 | 143.00 | 20.80 |
| 2.00 | 142.00 | 20.80 | 6.40 | 142.00 | 20.90 |
| 2.10 | 142.00 | 20.80 | 6.50 | 143.00 | 20.80 |
| 2.20 | 142.00 | 20.80 | 6.60 | 142.00 | 20.90 |
| 2.30 | 142.00 | 20.80 | 6.70 | 143.00 | 20.80 |
| 2.40 | 143.00 | 20.80 | 6.80 | 143.00 | 20.80 |
| 2.50 | 142.00 | 20.80 | 6.90 | 143.00 | 20.80 |
| 2.60 | 142.00 | 20.80 | 7.00 | 143.00 | 20.80 |
| 2.80 | 142.00 | 20.80 | 7.10 | 143.00 | 20.80 |
| 2.90 | 142.00 | 20.80 | 7.20 | 143.00 | 20.80 |
| 3.00 | 142.00 | 20.80 | 7.30 | 142.00 | 20.90 |
| 3.10 | 142.00 | 20.80 | 7.40 | 143.00 | 20.80 |
| 3.20 | 142.00 | 20.80 | 8.50 | 142.00 | 20.80 |
| 3.30 | 142.00 | 20.80 | 8.60 | 143.00 | 20.80 |
| 3.40 | 142.00 | 20.80 | 8.70 | 143.00 | 20.80 |
| 3.50 | 142.00 | 20.90 | 8.80 | 142.00 | 20.80 |
| 3.60 | 142.00 | 20.90 | | | |
| 3.70 | 142.00 | 20.90 | | | |
| 3.80 | 143.00 | 20.90 | | | |
| 3.90 | 142.00 | 20.90 | | | |
| 4.00 | 142.00 | 20.80 | | | |
| 4.10 | 142.00 | 20.80 | | | |
| 4.20 | 141.00 | 20.90 | | | |
| 4.30 | 142.00 | 20.90 | | | |
| 4.60 | 142.00 | 20.70 | | | |
| 4.70 | 142.00 | 20.90 | | | |
| 4.80 | 142.00 | 20.90 | | | |
| 4.90 | 143.00 | 20.90 | | | |
| 5.00 | 142.00 | 20.80 | | | |
| 5.10 | 142.00 | 20.90 | | | |
| 5.20 | 143.00 | 20.80 | | | |
| 5.30 | 142.00 | 20.80 | | | |
| 5.40 | 143.00 | 20.90 | | | |
| 5.50 | 142.00 | 20.80 | | | |

Table A-3

Experimental Results for Run # 02-SST

Initial Oxidation Temperature = 133 deg. C
Average System Pressure = 300 Psig.

Inlet Gas Composition = 21 % O₂
Average Gas Flow Rate = 300 cc/min

| Time, Hrs | Temp. °C | O ₂ Conc. % | Time, Hrs | Temp. °C | O ₂ Conc. % |
|-----------|----------|------------------------|-----------|----------|------------------------|
| 0.30 | 134.46 | 20.68 | 4.60 | 142.20 | 20.75 |
| 0.40 | 135.50 | 20.69 | 4.70 | 142.27 | 20.75 |
| 0.50 | 135.62 | 20.69 | 4.80 | 142.40 | 20.75 |
| 0.60 | 135.64 | 20.70 | 4.90 | 142.47 | 20.76 |
| 0.70 | 135.67 | 20.71 | 5.20 | 143.00 | 20.76 |
| 0.80 | 135.80 | 20.71 | 5.30 | 143.00 | 20.77 |
| 0.90 | 135.87 | 20.71 | 5.40 | 143.00 | 20.77 |
| 1.00 | 136.00 | 20.72 | 5.50 | 142.70 | 20.77 |
| 1.10 | 136.27 | 20.73 | 5.60 | 142.47 | 20.77 |
| 1.20 | 136.60 | 20.73 | 5.70 | 141.93 | 20.77 |
| 1.30 | 136.87 | 20.73 | 5.80 | 141.80 | 20.77 |
| 1.40 | 137.13 | 20.73 | 5.90 | 141.60 | 20.77 |
| 1.50 | 137.33 | 20.74 | 6.00 | 141.40 | 20.77 |
| 1.60 | 137.53 | 20.74 | 6.10 | 141.20 | 20.77 |
| 1.70 | 137.87 | 20.75 | 6.20 | 141.13 | 20.79 |
| 1.80 | 138.13 | 20.75 | 6.30 | 140.93 | 20.79 |
| 1.90 | 138.33 | 20.75 | 6.40 | 140.73 | 20.80 |
| 2.00 | 138.53 | 20.75 | 6.50 | 140.67 | 20.81 |
| 2.10 | 138.80 | 20.75 | 6.60 | 140.60 | 20.82 |
| 2.20 | 139.07 | 20.75 | 7.30 | 140.40 | 20.82 |
| 2.30 | 139.33 | 20.75 | 7.40 | 140.27 | 20.82 |
| 2.40 | 139.53 | 20.75 | 7.50 | 140.20 | 20.82 |
| 2.60 | 139.87 | 20.75 | 7.60 | 140.00 | 20.82 |
| 2.70 | 140.07 | 20.75 | 7.70 | 139.87 | 20.83 |
| 2.80 | 140.27 | 20.76 | 7.80 | 139.67 | 20.84 |
| 2.90 | 140.40 | 20.77 | 7.90 | 139.60 | 20.83 |
| 3.00 | 140.53 | 20.77 | 8.00 | 139.53 | 20.84 |
| 3.10 | 140.67 | 20.78 | 8.10 | 139.47 | 20.85 |
| 3.20 | 140.60 | 20.77 | 8.20 | 139.40 | 20.84 |
| 3.30 | 140.67 | 20.77 | 8.40 | 139.33 | 20.81 |
| 3.40 | 140.80 | 20.77 | 8.50 | 139.20 | 20.81 |
| 3.50 | 140.93 | 20.77 | 8.80 | 139.20 | 20.79 |
| 3.70 | 141.33 | 20.77 | 9.00 | 139.07 | 20.79 |
| 3.80 | 141.40 | 20.76 | 9.20 | 139.07 | 20.79 |
| 3.90 | 141.47 | 20.77 | 9.40 | 138.93 | 20.79 |
| 4.10 | 141.73 | 20.77 | 9.60 | 138.80 | 20.80 |
| 4.20 | 141.80 | 20.76 | 9.80 | 138.67 | 20.80 |
| 4.40 | 141.93 | 20.75 | 10.00 | 138.47 | 20.82 |
| 4.50 | 142.07 | 20.75 | 10.10 | 138.47 | 20.82 |

APPENDIX A-3 CONTD.

| | | |
|-------|--------|-------|
| 10.20 | 138.33 | 20.83 |
| 10.30 | 138.20 | 20.83 |
| 10.40 | 138.07 | 20.87 |
| 10.50 | 138.00 | 20.87 |
| 10.60 | 137.93 | 20.90 |
| 10.70 | 137.87 | 20.87 |
| 10.80 | 137.93 | 20.86 |
| 10.90 | 138.00 | 20.85 |
| 11.00 | 138.00 | 20.85 |
| 11.10 | 138.00 | 20.85 |
| 11.20 | 137.93 | 20.85 |
| 11.30 | 137.93 | 20.85 |
| 11.40 | 137.92 | 20.85 |
| 11.50 | 137.92 | 20.85 |
| 11.60 | 137.91 | 20.85 |
| 11.70 | 137.90 | 20.84 |
| 11.80 | 137.89 | 20.84 |
| 11.90 | 137.88 | 20.84 |

Table A-4

Experimental Results for Run # 03-SST

Initial Oxidation Temperature = 124 deg. C
Average System Pressure = 250 Psig.

Inlet Gas Composition = 21 % O₂
Average Gas Flow Rate = 240 cc/min

| Time, Hrs. | Temp. °C | O ₂ Conc. % | Time, Hrs. | Temp. °C | O ₂ Conc. % |
|------------|----------|------------------------|------------|----------|------------------------|
| 4.00 | 127.00 | 20.59 | 8.30 | 128.80 | 20.78 |
| 4.10 | 127.00 | 20.60 | 8.40 | 129.13 | 20.78 |
| 4.20 | 127.00 | 20.60 | 8.50 | 129.40 | 20.77 |
| 4.30 | 126.93 | 20.60 | 8.60 | 129.73 | 20.78 |
| 4.40 | 126.93 | 20.61 | 8.70 | 129.93 | 20.79 |
| 4.50 | 126.93 | 20.61 | 8.80 | 130.86 | 20.81 |
| 4.60 | 126.93 | 20.61 | 9.00 | 131.60 | 20.81 |
| 4.70 | 126.93 | 20.62 | 9.10 | 131.73 | 20.81 |
| 4.80 | 126.93 | 20.61 | 9.20 | 131.87 | 20.82 |
| 4.90 | 127.00 | 20.61 | 9.40 | 132.67 | 20.82 |
| 5.00 | 127.07 | 20.62 | 9.50 | 131.83 | 20.82 |
| 5.10 | 127.07 | 20.62 | 9.60 | 131.70 | 20.83 |
| 5.20 | 127.07 | 20.62 | 9.70 | 131.60 | 20.90 |
| 5.30 | 127.07 | 20.62 | 9.80 | 130.87 | 20.90 |
| 5.40 | 127.07 | 20.61 | 9.90 | 129.93 | 20.89 |
| 5.50 | 127.07 | 20.61 | 10.00 | 129.67 | 20.83 |
| 5.60 | 127.07 | 20.62 | 10.10 | 129.40 | 20.82 |
| 5.70 | 127.07 | 20.63 | 10.20 | 129.20 | 20.82 |
| 5.80 | 127.20 | 20.64 | 10.30 | 129.00 | 20.82 |
| 5.90 | 127.20 | 20.63 | 10.40 | 128.73 | 20.82 |
| 6.00 | 127.20 | 20.64 | 10.50 | 128.47 | 20.81 |
| 6.20 | 127.27 | 20.65 | 10.60 | 128.27 | 20.81 |
| 6.30 | 127.33 | 20.67 | 10.70 | 128.13 | 20.80 |
| 6.40 | 127.27 | 20.69 | 10.80 | 128.00 | 20.80 |
| 6.50 | 127.20 | 20.71 | 11.00 | 128.00 | 20.81 |
| 6.60 | 127.27 | 20.72 | 11.10 | 128.07 | 20.80 |
| 6.70 | 127.27 | 20.74 | 11.20 | 128.07 | 20.79 |
| 6.80 | 127.33 | 20.76 | 11.30 | 128.13 | 20.80 |
| 6.90 | 127.40 | 20.77 | 11.40 | 128.13 | 20.79 |
| 7.00 | 127.47 | 20.79 | 11.50 | 128.20 | 20.80 |
| 7.20 | 127.53 | 20.79 | 11.60 | 128.27 | 20.80 |
| 7.40 | 127.60 | 20.80 | 11.70 | 128.33 | 20.80 |
| 7.50 | 127.73 | 20.80 | 11.80 | 128.40 | 20.81 |
| 7.60 | 127.80 | 20.84 | 11.90 | 128.40 | 20.81 |
| 7.70 | 127.87 | 20.84 | 12.00 | 128.40 | 20.81 |
| 7.80 | 127.93 | 20.84 | 12.30 | 128.50 | 20.83 |
| 8.00 | 128.27 | 20.79 | 12.50 | 128.60 | 20.82 |
| 8.10 | 128.40 | 20.79 | 12.60 | 128.56 | 20.82 |
| 8.20 | 128.67 | 20.79 | 12.70 | 128.63 | 20.83 |

Table A-5

Experimental Results for Run # 04-SST

Initial Oxidation Temperature = 136 deg. C
Average System Pressure = 190 Psig.

Inlet Gas Composition = 21 % O₂
Average Gas Flow Rate = 180 cc/min

| Time, Hrs. | Temp. °C | O ₂ Conc. % | Time, Hrs. | Temp. °C | O ₂ Conc. % |
|------------|----------|------------------------|------------|----------|------------------------|
| 1.40 | 138.54 | 20.69 | 5.80 | 139.40 | 20.79 |
| 1.50 | 138.57 | 20.70 | 6.00 | 139.47 | 20.78 |
| 1.60 | 138.53 | 20.70 | 6.20 | 139.53 | 20.78 |
| 1.70 | 138.60 | 20.71 | 6.40 | 139.67 | 20.79 |
| 1.80 | 138.67 | 20.71 | 6.60 | 139.73 | 20.81 |
| 1.90 | 138.60 | 20.72 | 6.80 | 139.73 | 20.81 |
| 2.00 | 138.60 | 20.72 | 7.00 | 139.67 | 20.79 |
| 2.10 | 138.67 | 20.73 | 7.20 | 139.73 | 20.80 |
| 2.20 | 138.60 | 20.73 | 7.40 | 139.80 | 20.81 |
| 2.30 | 138.67 | 20.73 | 7.60 | 139.73 | 20.81 |
| 2.40 | 138.73 | 20.73 | 7.80 | 139.73 | 20.82 |
| 2.50 | 138.80 | 20.74 | 8.00 | 139.73 | 20.81 |
| 2.60 | 138.73 | 20.75 | 8.20 | 139.73 | 20.80 |
| 2.70 | 138.73 | 20.75 | 8.40 | 139.80 | 20.81 |
| 2.80 | 138.80 | 20.76 | 8.60 | 139.87 | 20.80 |
| 2.90 | 138.80 | 20.76 | 8.80 | 139.87 | 20.81 |
| 3.00 | 138.80 | 20.76 | 9.00 | 139.93 | 20.81 |
| 3.10 | 138.93 | 20.77 | 9.20 | 139.87 | 20.83 |
| 3.20 | 138.93 | 20.77 | 9.40 | 139.80 | 20.83 |
| 3.30 | 138.93 | 20.77 | 9.60 | 139.73 | 20.83 |
| 3.40 | 139.00 | 20.77 | 9.80 | 139.73 | 20.84 |
| 3.50 | 139.00 | 20.77 | 10.00 | 139.73 | 20.85 |
| 3.60 | 139.00 | 20.77 | 10.20 | 139.67 | 20.83 |
| 3.70 | 139.07 | 20.78 | 10.40 | 139.60 | 20.83 |
| 3.80 | 139.13 | 20.79 | 10.60 | 139.60 | 20.81 |
| 3.90 | 139.13 | 20.79 | 10.80 | 139.67 | 20.81 |
| 4.00 | 139.07 | 20.79 | 11.00 | 139.80 | 20.82 |
| 4.10 | 139.13 | 20.79 | 11.20 | 139.73 | 20.81 |
| 4.20 | 139.13 | 20.79 | 11.40 | 139.73 | 20.83 |
| 4.30 | 139.13 | 20.79 | 11.60 | 139.80 | 20.84 |
| 4.40 | 139.20 | 20.79 | 11.80 | 139.73 | 20.85 |
| 4.50 | 139.20 | 20.79 | 12.00 | 139.73 | 20.87 |
| 4.60 | 139.13 | 20.79 | 12.20 | 139.67 | 20.87 |
| 4.70 | 139.20 | 20.80 | 12.40 | 139.67 | 20.87 |
| 4.80 | 139.20 | 20.79 | 12.60 | 139.73 | 20.88 |
| 4.90 | 139.20 | 20.79 | 12.80 | 139.79 | 20.87 |
| 5.00 | 139.20 | 20.79 | 13.00 | 139.75 | 20.88 |
| 5.20 | 139.27 | 20.79 | 13.20 | 139.80 | 20.87 |
| 5.60 | 139.40 | 20.79 | 13.40 | 139.75 | 20.90 |

Table A-6

Experimental Results for Run # 05-SST

Initial Oxidation Temperature = 136 deg. C
Average System Pressure = 175 Psig.

Inlet Gas Composition = 21 % O₂
Average Gas Flow Rate = 100 cc/min

| Time, Hrs. | Temp. °C | O ₂ Conc. % | Time, Hrs. | Temp. °C | O ₂ Conc. % |
|------------|----------|------------------------|------------|----------|------------------------|
| 2.00 | 137.46 | 20.38 | 8.50 | 138.13 | 20.81 |
| 2.10 | 137.43 | 20.39 | 9.00 | 138.20 | 20.79 |
| 2.20 | 137.33 | 20.40 | 9.50 | 138.27 | 20.76 |
| 2.30 | 137.40 | 20.41 | 10.00 | 138.13 | 20.75 |
| 2.40 | 137.47 | 20.43 | 10.50 | 138.20 | 20.76 |
| 2.50 | 137.40 | 20.45 | 11.00 | 138.33 | 20.79 |
| 2.60 | 137.33 | 20.47 | 11.50 | 138.33 | 20.79 |
| 2.70 | 137.33 | 20.47 | 12.00 | 138.33 | 20.79 |
| 2.80 | 137.40 | 20.48 | 12.40 | 138.33 | 20.81 |
| 2.90 | 137.40 | 20.49 | 12.50 | 138.33 | 20.81 |
| 3.00 | 137.47 | 20.49 | 13.00 | 138.53 | 20.83 |
| 3.10 | 137.53 | 20.51 | 13.10 | 138.80 | 20.83 |
| 3.20 | 137.47 | 20.52 | 13.20 | 139.00 | 20.84 |
| 3.30 | 137.40 | 20.53 | 13.30 | 139.00 | 20.84 |
| 3.40 | 137.33 | 20.55 | 13.40 | 139.00 | 20.84 |
| 3.50 | 137.40 | 20.57 | 13.50 | 138.60 | 20.85 |
| 3.60 | 137.33 | 20.57 | 13.60 | 138.53 | 20.85 |
| 3.70 | 137.40 | 20.58 | 13.70 | 138.53 | 20.85 |
| 3.80 | 137.40 | 20.59 | 13.80 | 138.47 | 20.85 |
| 3.90 | 137.33 | 20.59 | 13.90 | 138.40 | 20.85 |
| 4.00 | 137.40 | 20.60 | 14.00 | 138.47 | 20.85 |
| 4.10 | 137.40 | 20.60 | 14.50 | 138.27 | 20.86 |
| 4.20 | 137.47 | 20.61 | 14.80 | 138.13 | 20.91 |
| 4.30 | 137.40 | 20.61 | | | |
| 4.40 | 137.40 | 20.63 | | | |
| 4.50 | 137.33 | 20.63 | | | |
| 4.60 | 137.33 | 20.64 | | | |
| 4.70 | 137.33 | 20.65 | | | |
| 4.80 | 137.40 | 20.65 | | | |
| 5.00 | 137.40 | 20.65 | | | |
| 5.10 | 137.47 | 20.67 | | | |
| 5.20 | 137.53 | 20.67 | | | |
| 5.30 | 137.53 | 20.67 | | | |
| 5.40 | 137.60 | 20.67 | | | |
| 6.00 | 137.87 | 20.69 | | | |
| 6.50 | 137.80 | 20.72 | | | |
| 7.00 | 137.87 | 20.71 | | | |
| 7.50 | 137.80 | 20.75 | | | |
| 8.00 | 137.93 | 20.77 | | | |

Table A-7

Experimental Results for Run # 06-SST

Initial Oxidation Temperature = 135 deg. C
Average System Pressure = 265 Psig.

Inlet Gas Composition = 40 % O₂
Average Gas Flow Rate = 420 cc/min

| Time, Hrs. | Temp. °C | O ₂ Conc. % | Time, Hrs. | Temp. °C | O ₂ Conc. % |
|------------|----------|------------------------|------------|----------|------------------------|
| 1.70 | 137.42 | 38.90 | 7.30 | 137.33 | 39.30 |
| 1.80 | 137.39 | 39.10 | 7.40 | 137.33 | 39.20 |
| 1.90 | 137.36 | 39.10 | 7.50 | 137.33 | 39.30 |
| 2.20 | 137.33 | 39.10 | 7.60 | 137.33 | 39.40 |
| 2.40 | 137.40 | 38.70 | 7.70 | 137.47 | 39.30 |
| 2.50 | 137.40 | 38.20 | 7.80 | 137.60 | 39.20 |
| 2.60 | 137.47 | 37.90 | 7.90 | 137.67 | 39.40 |
| 2.90 | 137.47 | 36.70 | 8.00 | 137.67 | 39.30 |
| 3.10 | 137.53 | 35.60 | 8.10 | 137.73 | 39.40 |
| 3.20 | 137.53 | 35.40 | 8.20 | 137.87 | 39.40 |
| 3.30 | 137.60 | 39.20 | 8.30 | 137.87 | 39.30 |
| 3.40 | 137.73 | 39.10 | 8.40 | 137.93 | 39.40 |
| 3.60 | 137.73 | 39.30 | 8.50 | 138.00 | 39.30 |
| 3.70 | 137.87 | 39.30 | 8.60 | 138.07 | 39.40 |
| 4.00 | 137.93 | 39.30 | 8.70 | 138.07 | 39.40 |
| 4.20 | 137.93 | 39.30 | 8.80 | 138.07 | 39.30 |
| 4.30 | 137.93 | 39.20 | 8.90 | 138.00 | 39.30 |
| 4.40 | 137.93 | 39.10 | 9.00 | 138.00 | 39.30 |
| 4.50 | 137.93 | 39.20 | 9.10 | 138.00 | 39.30 |
| 4.60 | 137.93 | 39.30 | 9.20 | 137.93 | 39.40 |
| 4.80 | 137.87 | 39.10 | 9.30 | 137.93 | 39.40 |
| 4.90 | 137.73 | 39.30 | 9.40 | 137.87 | 39.30 |
| 5.00 | 137.67 | 39.10 | 9.50 | 137.93 | 39.30 |
| 5.10 | 137.60 | 39.30 | 9.60 | 137.87 | 39.40 |
| 5.20 | 137.60 | 39.40 | 9.70 | 137.73 | 39.30 |
| 5.30 | 137.53 | 39.40 | 9.80 | 137.87 | 39.40 |
| 5.40 | 137.47 | 39.30 | 9.90 | 137.80 | 39.40 |
| 5.60 | 137.47 | 39.10 | 10.00 | 137.80 | 39.30 |
| 5.70 | 137.33 | 39.10 | 10.10 | 137.73 | 39.30 |
| 5.80 | 137.27 | 39.30 | 10.20 | 137.73 | 39.30 |
| 6.00 | 137.20 | 39.40 | 10.30 | 137.73 | 39.40 |
| 6.30 | 137.20 | 39.30 | 10.40 | 137.80 | 39.30 |
| 6.60 | 137.20 | 39.40 | 10.50 | 137.73 | 39.30 |
| 6.70 | 137.27 | 39.30 | 10.60 | 137.80 | 39.20 |
| 6.80 | 137.27 | 39.40 | 10.70 | 137.80 | 39.30 |
| 6.90 | 137.27 | 39.30 | 10.80 | 137.73 | 39.30 |
| 7.00 | 137.27 | 39.30 | 10.90 | 137.73 | 39.30 |
| 7.10 | 137.27 | 39.30 | 11.00 | 137.73 | 39.40 |
| 7.20 | 137.33 | 39.40 | 11.10 | 137.80 | 39.30 |

Table A-7 contd.

| Time, Hrs. | Temp. °C | O ₂ Conc. % |
|------------|----------|------------------------|
| 11.20 | 137.73 | 39.30 |
| 11.30 | 137.67 | 39.40 |
| 11.40 | 137.73 | 39.30 |
| 11.50 | 137.73 | 39.30 |
| 11.60 | 137.80 | 39.30 |
| 11.70 | 137.73 | 39.40 |
| 11.80 | 137.73 | 39.40 |
| 11.90 | 137.73 | 39.40 |
| 12.00 | 137.73 | 39.30 |
| 12.10 | 137.80 | 39.30 |
| 12.20 | 137.80 | 39.30 |
| 12.30 | 137.79 | 39.40 |
| 12.40 | 137.85 | 39.30 |
| 12.50 | 137.83 | 39.30 |
| 12.60 | 137.82 | 39.40 |
| 12.70 | 137.90 | 39.30 |
| 12.80 | 137.89 | 39.30 |
| 12.90 | 137.88 | 39.40 |

Table A-8

Experimental Results for Run # 07-SST

Initial Oxidation Temperature = 135 deg. C
Average System Pressure = 450 Psig.

Inlet Gas Composition = 40 % O₂
Average Gas Flow Rate = - cc/min

| Time, Hrs. | Temp. °C | O ₂ Conc. % | Time, Hrs. | Temp. °C | O ₂ Conc. % |
|------------|----------|------------------------|------------|----------|------------------------|
| 2.20 | 138.17 | 36.56 | 8.40 | 137.00 | 36.64 |
| 2.30 | 138.23 | 36.60 | 8.50 | 137.00 | 36.63 |
| 2.40 | 138.21 | 36.61 | 8.60 | 137.00 | 36.64 |
| 2.50 | 138.20 | 36.61 | 8.70 | 137.70 | 36.60 |
| 2.60 | 138.33 | 36.64 | 8.80 | 137.73 | 36.69 |
| 3.00 | 138.40 | 36.63 | 8.90 | 137.80 | 36.64 |
| 3.10 | 138.47 | 36.61 | 9.00 | 137.80 | 36.67 |
| 3.20 | 138.40 | 36.57 | 9.10 | 137.87 | 36.65 |
| 3.30 | 138.33 | 36.57 | 9.20 | 137.87 | 36.66 |
| 3.40 | 138.40 | 36.59 | 9.30 | 137.93 | 36.71 |
| 3.50 | 138.47 | 36.59 | 9.40 | 137.93 | 36.67 |
| 3.60 | 138.53 | 36.65 | 9.50 | 137.93 | 36.67 |
| 3.70 | 138.60 | 36.63 | 9.60 | 137.93 | 36.73 |
| 3.80 | 138.60 | 36.59 | 9.70 | 138.00 | 36.69 |
| 3.90 | 138.67 | 36.60 | 9.80 | 138.00 | 36.71 |
| 4.00 | 138.67 | 36.66 | 9.90 | 137.93 | 36.71 |
| 5.10 | 138.67 | 36.65 | 10.00 | 137.93 | 36.70 |
| 5.80 | 138.73 | 36.64 | 10.10 | 137.93 | 36.75 |
| 5.90 | 138.80 | 36.60 | 10.20 | 137.93 | 36.73 |
| 6.00 | 139.00 | 36.59 | 10.30 | 138.00 | 36.63 |
| 6.10 | 139.00 | 36.59 | 10.40 | 138.00 | 36.70 |
| 6.30 | 139.00 | 36.65 | 10.50 | 138.00 | 36.73 |
| 6.40 | 138.87 | 36.67 | 10.60 | 138.00 | 36.78 |
| 6.50 | 138.87 | 36.70 | 11.00 | 138.00 | 36.81 |
| 6.60 | 138.80 | 36.67 | 11.10 | 138.00 | 36.79 |
| 6.70 | 138.73 | 36.66 | 12.00 | 138.00 | 36.80 |
| 6.80 | 138.67 | 36.61 | 12.10 | 138.00 | 36.81 |
| 6.90 | 138.60 | 36.67 | 12.20 | 138.00 | 36.74 |
| 7.00 | 138.47 | 36.63 | 12.30 | 138.00 | 36.84 |
| 7.10 | 138.33 | 36.63 | 12.40 | 138.07 | 36.78 |
| 7.20 | 138.27 | 36.59 | 12.50 | 138.13 | 36.76 |
| 7.60 | 138.13 | 36.57 | 12.60 | 138.13 | 36.81 |
| 7.70 | 138.07 | 36.57 | 12.70 | 138.20 | 36.78 |
| 7.80 | 137.93 | 36.61 | 12.80 | 138.20 | 36.80 |
| 7.90 | 137.87 | 36.67 | 12.90 | 138.20 | 36.90 |
| 8.00 | 137.80 | 36.67 | 13.00 | 138.20 | 36.81 |
| 8.10 | 137.73 | 36.62 | 13.10 | 138.20 | 36.75 |
| 8.20 | 137.60 | 36.62 | 13.20 | 138.27 | 36.71 |
| 8.30 | 137.60 | 36.61 | 13.30 | 138.27 | 36.65 |

Tabel A-8 Contd.

| Time, Hrs. | Temp. °C | O ₂ Conc. % |
|------------|----------|------------------------|
| 13.40 | 139.00 | 36.61 |
| 13.50 | 139.00 | 36.61 |
| 13.60 | 139.00 | 36.63 |
| 13.70 | 138.80 | 36.71 |
| 13.80 | 138.83 | 36.64 |
| 13.90 | 138.89 | 36.68 |
| 14.00 | 138.73 | 36.69 |
| 14.10 | 138.66 | 36.67 |
| 14.20 | 138.30 | 36.66 |
| 14.30 | 138.33 | 36.64 |
| 14.40 | 138.38 | 36.58 |

Table A-9

Experimental Results for Run # 08-SST

Initial Oxidation Temperature = 134 deg. C
Average System Pressure = 240 Psig.

Inlet Gas Composition = 40 % O₂
Average Gas Flow Rate = 400 cc/min

| Time, Hrs. | Temp. °C | O ₂ Conc. % | Time, Hrs. | Temp. °C | O ₂ Conc. % |
|------------|----------|------------------------|------------|----------|------------------------|
| 1.25 | 134.30 | 39.36 | 1.90 | 136.60 | 39.49 |
| 1.27 | 134.91 | 39.35 | 1.92 | 136.53 | 39.49 |
| 1.28 | 134.83 | 39.37 | 1.93 | 136.47 | 39.49 |
| 1.30 | 134.85 | 39.36 | 1.98 | 136.60 | 39.49 |
| 1.32 | 134.86 | 39.37 | 2.00 | 136.67 | 39.50 |
| 1.33 | 134.87 | 39.37 | 2.02 | 136.67 | 39.51 |
| 1.35 | 134.87 | 39.37 | 2.05 | 136.73 | 39.50 |
| 1.37 | 134.80 | 39.39 | 2.08 | 136.73 | 39.49 |
| 1.38 | 134.87 | 39.39 | 2.10 | 136.73 | 39.49 |
| 1.40 | 134.87 | 39.40 | 2.12 | 136.73 | 39.49 |
| 1.42 | 134.87 | 39.41 | 2.15 | 136.80 | 39.49 |
| 1.43 | 134.87 | 39.41 | 2.17 | 136.87 | 39.49 |
| 1.45 | 134.87 | 39.42 | 2.18 | 136.93 | 39.49 |
| 1.47 | 134.93 | 39.43 | 2.20 | 136.87 | 39.49 |
| 1.48 | 135.07 | 39.42 | 2.22 | 136.93 | 39.49 |
| 1.50 | 135.20 | 39.43 | 2.25 | 136.87 | 39.49 |
| 1.52 | 135.33 | 39.43 | 2.28 | 136.87 | 39.47 |
| 1.53 | 135.47 | 39.43 | 2.30 | 136.87 | 39.48 |
| 1.55 | 135.60 | 39.44 | 2.32 | 136.87 | 39.49 |
| 1.57 | 135.67 | 39.44 | 2.38 | 136.80 | 39.51 |
| 1.58 | 135.80 | 39.45 | 2.40 | 136.80 | 39.51 |
| 1.60 | 135.87 | 39.45 | 2.60 | 136.73 | 39.52 |
| 1.62 | 136.00 | 39.45 | 2.80 | 137.07 | 39.53 |
| 1.63 | 136.07 | 39.46 | 3.00 | 136.93 | 39.54 |
| 1.65 | 136.20 | 39.46 | 3.20 | 136.73 | 39.53 |
| 1.67 | 136.33 | 39.46 | 3.40 | 136.80 | 39.53 |
| 1.68 | 136.47 | 39.46 | 3.60 | 137.00 | 39.58 |
| 1.70 | 136.53 | 39.46 | 3.80 | 136.93 | 39.57 |
| 1.72 | 136.60 | 39.46 | 4.00 | 137.00 | 39.58 |
| 1.73 | 136.53 | 39.47 | 4.20 | 137.00 | 39.55 |
| 1.75 | 136.47 | 39.47 | 4.40 | 137.00 | 39.57 |
| 1.77 | 136.47 | 39.48 | 4.60 | 137.07 | 39.57 |
| 1.78 | 136.53 | 39.49 | 4.80 | 137.47 | 39.59 |
| 1.80 | 136.47 | 39.49 | 5.00 | 137.20 | 39.57 |
| 1.82 | 136.53 | 39.49 | 5.20 | 137.27 | 39.59 |
| 1.83 | 136.47 | 39.48 | 5.40 | 137.40 | 39.58 |
| 1.85 | 136.53 | 39.49 | 5.60 | 137.33 | 39.57 |
| 1.87 | 136.60 | 39.49 | 5.80 | 137.33 | 39.61 |
| 1.88 | 136.67 | 39.49 | 6.00 | 137.13 | 39.61 |

Table A-9 contd.

| Time, Hrs. | Temp. °C | O ₂ Conc. % |
|------------|----------|------------------------|
| 6.20 | 137.33 | 39.60 |
| 6.40 | 137.33 | 39.59 |
| 6.60 | 137.27 | 39.57 |
| 6.80 | 137.33 | 39.59 |
| 7.00 | 137.40 | 39.61 |
| 7.20 | 137.40 | 39.61 |
| 7.40 | 137.53 | 39.58 |
| 7.60 | 137.33 | 39.61 |
| 7.80 | 137.73 | 39.58 |
| 8.00 | 137.60 | 39.58 |
| 8.20 | 137.67 | 39.62 |
| 8.40 | 137.47 | 39.61 |
| 8.60 | 137.67 | 39.61 |
| 8.80 | 137.53 | 39.62 |
| 9.00 | 137.80 | 39.61 |
| 9.20 | 137.80 | 39.65 |
| 9.40 | 137.67 | 39.63 |
| 9.60 | 137.73 | 39.61 |
| 9.80 | 138.07 | 39.63 |
| 10.00 | 138.00 | 39.61 |
| 10.20 | 137.93 | 39.61 |
| 10.40 | 137.87 | 39.67 |
| 10.60 | 137.80 | 39.63 |
| 10.80 | 137.60 | 39.62 |
| 11.00 | 137.93 | 39.63 |
| 11.20 | 137.80 | 39.63 |
| 11.40 | 137.87 | 39.67 |
| 11.60 | 137.93 | 39.65 |
| 11.80 | 137.73 | 39.68 |
| 12.00 | 137.80 | 39.65 |
| 12.20 | 137.80 | 39.65 |
| 12.40 | 137.87 | 39.65 |
| 12.42 | 137.80 | 39.64 |
| 12.43 | 137.80 | 39.65 |
| 12.45 | 137.86 | 39.64 |
| 12.47 | 137.85 | 39.65 |
| 12.48 | 137.83 | 39.65 |
| 12.50 | 137.82 | 39.65 |
| 12.52 | 137.70 | 39.65 |
| 12.53 | 137.78 | 39.66 |
| 12.55 | 137.75 | 39.66 |

Table A-10

Experimental Results for Run # 01-BT

Initial Oxidation Temperature = 141 deg. C
Average System Pressure = 260 Psig.

Inlet Gas Composition = 40 % O₂
Average Gas Flow Rate = 370 cc/min

| Time, Hrs. | Temp. °C | O ₂ Conc. % | Time, Hrs. | Temp. °C | O ₂ Conc. % |
|------------|----------|------------------------|------------|----------|------------------------|
| 1.60 | 135.00 | 39.30 | 8.20 | 136.00 | 39.40 |
| 1.65 | 135.00 | 39.20 | 8.40 | 135.00 | 39.30 |
| 1.70 | 134.00 | 39.30 | 8.60 | 135.00 | 39.40 |
| 1.75 | 135.00 | 39.30 | 8.80 | 135.00 | 39.40 |
| 1.80 | 135.00 | 39.30 | 9.00 | 136.00 | 39.40 |
| 1.85 | 134.00 | 39.30 | 9.20 | 135.00 | 39.30 |
| 1.90 | 135.00 | 39.30 | 9.40 | 135.00 | 39.50 |
| 1.95 | 136.00 | 39.30 | 9.60 | 136.00 | 39.30 |
| 2.00 | 134.00 | 39.30 | 9.80 | 136.00 | 39.40 |
| 2.20 | 135.00 | 39.30 | 10.00 | 136.00 | 39.40 |
| 2.40 | 135.00 | 39.30 | 10.20 | 135.00 | 39.50 |
| 2.60 | 135.00 | 39.30 | 10.40 | 136.00 | 39.40 |
| 2.80 | 135.00 | 39.20 | 10.60 | 136.00 | 39.50 |
| 3.00 | 135.00 | 39.30 | 10.80 | 136.00 | 39.40 |
| 3.20 | 135.00 | 39.30 | 11.00 | 136.00 | 39.50 |
| 3.40 | 135.00 | 39.50 | 11.20 | 135.00 | 39.40 |
| 3.60 | 135.00 | 39.50 | 11.40 | 135.00 | 39.40 |
| 3.80 | 135.00 | 39.50 | 11.60 | 135.00 | 39.40 |
| 4.00 | 136.00 | 39.50 | 11.80 | 135.00 | 39.40 |
| 4.20 | 135.00 | 39.50 | 12.00 | 135.00 | 39.40 |
| 4.40 | 134.00 | 39.50 | 12.20 | 136.00 | 39.50 |
| 4.60 | 135.00 | 39.40 | 12.25 | 135.00 | 39.40 |
| 4.80 | 135.00 | 39.40 | 12.30 | 135.00 | 39.50 |
| 5.00 | 135.00 | 39.50 | | | |
| 5.20 | 135.00 | 39.50 | | | |
| 5.40 | 136.00 | 39.40 | | | |
| 5.60 | 134.00 | 39.50 | | | |
| 5.80 | 134.00 | 39.50 | | | |
| 6.00 | 135.00 | 39.40 | | | |
| 6.20 | 135.00 | 39.50 | | | |
| 6.40 | 134.00 | 39.40 | | | |
| 6.60 | 135.00 | 39.40 | | | |
| 6.80 | 135.00 | 39.40 | | | |
| 7.00 | 135.00 | 39.40 | | | |
| 7.20 | 135.00 | 39.30 | | | |
| 7.40 | 135.00 | 39.40 | | | |
| 7.60 | 135.00 | 39.30 | | | |
| 7.80 | 135.00 | 39.40 | | | |
| 8.00 | 135.00 | 39.40 | | | |

Table A-11

Experimental Results for Run # 02-BT

Initial Oxidation Temperature = 133 deg. C Inlet Gas Composition = 40 % O₂
 Average System Pressure = 1050 Psig. Average Gas Flow Rate = - cc/min

| Time, Hrs. | Temp. °C | O ₂ Conc. % | Time, Hrs. | Temp. °C | O ₂ Conc. % |
|------------|----------|------------------------|------------|----------|------------------------|
| 1.30 | 136.25 | 36.19 | 10.50 | 137.00 | 38.43 |
| 1.40 | 136.31 | 36.13 | 10.60 | 137.07 | 37.01 |
| 1.60 | 136.36 | 36.19 | 10.70 | 137.00 | 36.96 |
| 1.80 | 136.27 | 36.18 | 10.80 | 137.07 | 37.01 |
| 1.90 | 136.20 | 36.19 | 11.70 | 137.00 | 37.05 |
| 2.00 | 136.20 | 36.26 | 13.20 | 136.93 | 37.02 |
| 2.20 | 136.13 | 36.21 | 13.30 | 136.93 | 36.99 |
| 2.30 | 136.13 | 36.21 | 13.40 | 136.80 | 36.79 |
| 2.40 | 136.07 | 36.15 | 13.50 | 136.67 | 36.58 |
| 2.60 | 136.00 | 36.13 | 13.60 | 136.53 | 36.59 |
| 3.00 | 136.00 | 36.17 | 13.70 | 136.40 | 36.59 |
| 3.10 | 135.93 | 36.18 | 13.80 | 136.40 | 36.61 |
| 3.60 | 135.87 | 36.18 | 13.90 | 136.33 | 36.61 |
| 3.70 | 135.87 | 36.06 | 14.00 | 136.40 | 36.61 |
| 3.80 | 135.80 | 36.13 | 14.10 | 136.40 | 36.63 |
| 3.90 | 135.73 | 36.19 | 14.20 | 136.40 | 36.67 |
| 4.10 | 135.67 | 36.25 | 14.30 | 136.40 | 36.62 |
| 4.20 | 135.60 | 36.22 | 14.40 | 136.47 | 36.65 |
| 4.30 | 134.00 | 36.29 | 14.50 | 136.47 | 36.61 |
| 4.40 | 134.00 | 36.33 | 14.60 | 136.53 | 36.60 |
| 4.50 | 134.00 | 36.35 | 14.70 | 136.60 | 36.74 |
| 4.60 | 134.00 | 36.42 | 14.80 | 136.67 | 36.91 |
| 4.70 | 134.00 | 36.41 | 14.90 | 136.73 | 36.93 |
| 4.80 | 135.73 | 36.42 | 15.00 | 136.80 | 36.59 |
| 4.90 | 135.80 | 36.47 | 15.10 | 136.87 | 36.53 |
| 5.00 | 135.87 | 36.48 | 15.20 | 136.93 | 36.87 |
| 5.10 | 135.87 | 36.46 | 15.30 | 136.93 | 36.88 |
| 5.30 | 136.00 | 36.47 | 15.40 | 137.00 | 36.91 |
| 5.40 | 136.07 | 36.64 | 15.50 | 137.07 | 36.87 |
| 5.50 | 136.07 | 36.65 | 15.60 | 137.07 | 36.88 |
| 5.60 | 136.20 | 36.81 | 15.70 | 137.07 | 36.91 |
| 5.80 | 136.33 | 37.03 | 15.80 | 137.07 | 36.99 |
| 5.90 | 136.47 | 37.05 | 15.90 | 137.08 | 37.02 |
| 8.30 | 136.60 | 37.03 | 16.00 | 137.08 | 37.05 |
| 9.30 | 136.73 | 36.99 | 16.10 | 137.09 | 37.07 |
| 9.40 | 136.80 | 36.97 | 16.50 | 137.10 | 36.93 |
| 9.50 | 136.87 | 36.97 | 17.90 | 137.11 | 36.71 |
| 10.30 | 136.93 | 38.53 | 18.00 | 137.13 | 36.70 |

Table A-12

Experimental Results for Run # 01-LT

Initial Oxidation Temperature = 111deg. C
Average System Pressure = 600 Psig.

Inlet Gas Composition = 40 % O₂
Average Gas Flow Rate = 250 cc/min

| Time, Hrs. | Temp. °C | O ₂ Conc. % | Time, Hrs. | Temp. °C | O ₂ Conc. % |
|------------|----------|------------------------|------------|----------|------------------------|
| 2.85 | 113.00 | 39.10 | 5.20 | 113.00 | 37.70 |
| 2.90 | 113.00 | 38.90 | 5.25 | 113.00 | 37.60 |
| 2.95 | 112.00 | 38.90 | 5.30 | 113.00 | 37.50 |
| 3.00 | 111.00 | 39.00 | 5.35 | 113.00 | 37.60 |
| 3.05 | 113.00 | 38.90 | 5.40 | 113.00 | 37.60 |
| 3.10 | 111.00 | 39.00 | 5.45 | 113.00 | 37.50 |
| 3.15 | 112.00 | 38.90 | 5.50 | 113.00 | 37.60 |
| 3.20 | 112.00 | 39.00 | 5.55 | 113.00 | 37.50 |
| 3.25 | 112.00 | 39.00 | 5.60 | 113.00 | 37.50 |
| 3.30 | 112.00 | 39.00 | 5.65 | 113.00 | 37.50 |
| 3.35 | 112.00 | 39.00 | 5.70 | 113.00 | 37.50 |
| 3.40 | 112.00 | 38.90 | 5.75 | 113.00 | 37.60 |
| 3.45 | 112.00 | 39.00 | 5.80 | 113.00 | 37.60 |
| 3.50 | 112.00 | 39.00 | 5.85 | 113.00 | 37.60 |
| 3.55 | 112.00 | 39.00 | 5.90 | 113.00 | 37.60 |
| 3.65 | 112.00 | 39.10 | 5.95 | 114.00 | 37.60 |
| 3.70 | 112.00 | 39.00 | 6.00 | 112.00 | 37.60 |
| 3.75 | 112.00 | 39.00 | 6.05 | 113.00 | 37.60 |
| 3.80 | 112.00 | 38.90 | 6.10 | 113.00 | 37.60 |
| 3.85 | 112.00 | 39.00 | 6.15 | 113.00 | 37.60 |
| 3.90 | 112.00 | 38.90 | 6.20 | 113.00 | 37.70 |
| 3.95 | 113.00 | 38.90 | 6.25 | 114.00 | 37.70 |
| 4.00 | 112.00 | 38.90 | 6.30 | 113.00 | 37.70 |
| 4.05 | 113.00 | 38.80 | 6.35 | 114.00 | 37.80 |
| 4.10 | 112.00 | 38.80 | 6.40 | 113.00 | 37.80 |
| 4.15 | 113.00 | 38.70 | 6.45 | 113.00 | 37.80 |
| 4.20 | 112.00 | 38.60 | 6.50 | 113.00 | 37.80 |
| 4.35 | 113.00 | 38.50 | 6.55 | 113.00 | 37.90 |
| 4.40 | 112.00 | 38.40 | 6.60 | 113.00 | 37.90 |
| 4.45 | 112.00 | 38.40 | 6.65 | 113.00 | 37.90 |
| 4.50 | 112.00 | 38.30 | 6.70 | 113.00 | 38.00 |
| 4.65 | 113.00 | 38.10 | 6.75 | 113.00 | 38.00 |
| 4.70 | 112.00 | 38.00 | 6.80 | 113.00 | 38.00 |
| 4.75 | 113.00 | 38.00 | 6.85 | 112.00 | 38.00 |
| 4.80 | 113.00 | 37.90 | 6.90 | 112.00 | 38.00 |
| 4.85 | 112.00 | 37.90 | 7.10 | 113.00 | 38.00 |
| 4.90 | 112.00 | 37.80 | 7.15 | 113.00 | 38.10 |
| 4.95 | 113.00 | 37.80 | 7.20 | 113.00 | 38.10 |
| 5.00 | 112.00 | 37.80 | 7.25 | 113.00 | 38.10 |
| 5.05 | 113.00 | 37.70 | 7.30 | 113.00 | 38.10 |

Table A-12 Contd

| Time, Hrs. | Temp.°C | O ₂ Conc. % |
|------------|---------|------------------------|
| 7.35 | 113.00 | 38.10 |
| 7.40 | 113.00 | 38.00 |
| 7.45 | 113.00 | 38.10 |
| 7.55 | 113.00 | 38.00 |
| 7.95 | 113.00 | 38.90 |
| 8.15 | 113.00 | 38.70 |
| 9.20 | 114.00 | 38.70 |
| 9.40 | 113.00 | 38.70 |
| 9.60 | 114.00 | 38.70 |
| 9.80 | 114.00 | 38.70 |
| 10.00 | 114.00 | 38.70 |
| 10.20 | 113.00 | 38.70 |
| 10.40 | 114.00 | 38.70 |
| 10.60 | 113.00 | 38.70 |

Table A-13

Experimental Results for Run # 02-LT

Initial Oxidation Temperature = 122 deg. C
Average System Pressure = 625 Psig.

Inlet Gas Composition = 40 % O₂
Average Gas Flow Rate = 250 cc/min

| Time, Hrs. | Temp. °C | O ₂ Conc. % | Time, Hrs. | Temp. °C | O ₂ Conc. % |
|------------|----------|------------------------|------------|----------|------------------------|
| 0.10 | 125.63 | 36.53 | 4.70 | 137.87 | 35.45 |
| 0.20 | 129.09 | 35.81 | 4.80 | 137.87 | 35.45 |
| 0.50 | 133.10 | 34.47 | 4.90 | 138.00 | 35.47 |
| 0.60 | 133.27 | 34.67 | 5.00 | 138.00 | 35.53 |
| 0.70 | 133.42 | 35.14 | 5.10 | 138.00 | 35.51 |
| 0.90 | 133.69 | 35.18 | 5.40 | 138.00 | 35.54 |
| 1.00 | 133.86 | 35.20 | 5.50 | 138.00 | 35.55 |
| 1.10 | 134.07 | 35.19 | 5.60 | 138.07 | 35.57 |
| 1.30 | 134.87 | 35.13 | 5.70 | 138.13 | 35.52 |
| 1.40 | 135.40 | 35.08 | 5.80 | 138.13 | 35.52 |
| 1.50 | 135.47 | 35.07 | 6.80 | 138.13 | 35.57 |
| 1.60 | 135.53 | 35.09 | 6.90 | 138.13 | 35.60 |
| 1.70 | 135.67 | 35.14 | 7.00 | 138.13 | 35.62 |
| 1.80 | 135.80 | 35.13 | 7.10 | 138.07 | 35.64 |
| 1.90 | 135.87 | 35.08 | 7.30 | 138.13 | 35.72 |
| 2.00 | 135.93 | 35.12 | 7.40 | 138.13 | 35.72 |
| 2.10 | 136.07 | 35.09 | 7.50 | 138.13 | 35.75 |
| 2.20 | 136.20 | 35.14 | 8.00 | 138.20 | 35.79 |
| 2.30 | 136.27 | 35.14 | 8.10 | 138.20 | 35.81 |
| 2.40 | 136.33 | 35.19 | 8.20 | 138.20 | 35.89 |
| 2.50 | 136.27 | 35.15 | 8.30 | 138.20 | 35.87 |
| 2.70 | 136.33 | 35.15 | 8.40 | 138.27 | 35.91 |
| 2.90 | 136.27 | 35.14 | 8.50 | 138.20 | 35.93 |
| 3.00 | 136.27 | 35.15 | 8.90 | 138.13 | 36.02 |
| 3.10 | 136.27 | 35.22 | 9.00 | 138.20 | 36.09 |
| 3.30 | 136.40 | 35.30 | 9.10 | 138.20 | 36.09 |
| 3.40 | 136.40 | 35.33 | 9.20 | 138.27 | 36.10 |
| 3.50 | 136.53 | 35.31 | 9.30 | 138.27 | 36.11 |
| 3.60 | 136.67 | 35.37 | 9.40 | 138.27 | 36.11 |
| 3.70 | 136.80 | 35.44 | 9.80 | 138.27 | 36.16 |
| 3.80 | 136.93 | 35.42 | 9.90 | 138.27 | 36.16 |
| 3.90 | 137.00 | 35.47 | 10.00 | 138.27 | 36.15 |
| 4.00 | 137.07 | 35.48 | 10.10 | 138.27 | 36.16 |
| 4.10 | 137.13 | 35.49 | 10.20 | 138.33 | 36.23 |
| 4.20 | 137.27 | 35.48 | 10.40 | 138.27 | 36.20 |
| 4.30 | 137.40 | 35.49 | 11.80 | 138.27 | 36.25 |
| 4.40 | 137.47 | 35.48 | 11.90 | 138.13 | 36.34 |
| 4.50 | 137.67 | 35.51 | 12.00 | 138.07 | 36.35 |
| 4.60 | 137.80 | 35.44 | 12.10 | 138.00 | 36.31 |

Table A-13 Contd.

| Time, Hrs. | Temp. °C | O ₂ Conc. % | Time, Hrs. | Temp. °C | O ₂ Conc. % |
|------------|----------|------------------------|------------|----------|------------------------|
| 12.20 | 137.93 | 36.32 | 20.00 | 138.80 | 36.81 |
| 12.30 | 137.93 | 36.32 | 20.10 | 138.60 | 36.79 |
| 12.40 | 137.80 | 36.35 | 20.20 | 138.47 | 36.75 |
| 12.50 | 137.73 | 36.45 | 20.30 | 138.40 | 36.78 |
| 12.90 | 137.67 | 36.52 | 20.40 | 138.27 | 36.83 |
| 14.90 | 137.60 | 36.51 | 20.50 | 138.13 | 36.89 |
| 15.00 | 137.53 | 36.58 | 20.60 | 138.13 | 36.97 |
| 15.90 | 137.47 | 36.69 | 20.70 | 138.07 | 36.94 |
| 16.00 | 137.33 | 36.71 | 20.80 | 138.07 | 36.99 |
| 16.10 | 137.27 | 36.67 | 21.00 | 138.07 | 37.05 |
| 16.20 | 137.27 | 36.71 | 21.10 | 138.13 | 37.09 |
| 16.30 | 137.20 | 36.70 | 21.20 | 138.13 | 37.07 |
| 16.40 | 137.20 | 36.70 | 21.70 | 138.20 | 37.02 |
| 16.50 | 137.20 | 36.75 | 21.80 | 138.27 | 36.93 |
| 16.60 | 137.27 | 36.82 | 21.90 | 138.33 | 36.97 |
| 16.80 | 137.33 | 36.81 | 22.00 | 138.40 | 36.97 |
| 17.00 | 137.33 | 36.86 | 22.10 | 138.47 | 36.99 |
| 17.10 | 137.33 | 36.95 | 22.20 | 138.53 | 37.01 |
| 17.20 | 137.33 | 36.95 | 22.30 | 138.53 | 36.99 |
| 17.30 | 137.33 | 36.96 | 22.40 | 138.67 | 36.93 |
| 17.40 | 137.27 | 37.01 | 22.50 | 138.67 | 36.91 |
| 17.50 | 137.33 | 37.00 | 22.60 | 138.67 | 36.93 |
| 17.60 | 137.40 | 36.95 | 22.70 | 138.60 | 36.87 |
| 17.70 | 137.47 | 36.95 | 22.80 | 138.60 | 36.89 |
| 17.80 | 137.47 | 36.93 | 22.90 | 138.53 | 36.83 |
| 17.90 | 137.60 | 36.97 | 23.00 | 138.47 | 36.81 |
| 18.00 | 137.67 | 36.99 | 23.10 | 138.47 | 36.85 |
| 18.10 | 137.73 | 36.95 | 23.20 | 138.33 | 36.81 |
| 18.20 | 137.87 | 36.95 | 23.30 | 138.27 | 36.84 |
| 18.30 | 137.93 | 36.93 | 23.40 | 138.20 | 36.76 |
| 18.40 | 138.07 | 36.98 | 23.50 | 138.20 | 36.77 |
| 18.50 | 138.20 | 36.99 | 23.60 | 138.13 | 36.79 |
| 18.60 | 138.33 | 36.92 | 23.70 | 138.07 | 36.78 |
| 18.70 | 138.53 | 36.86 | 23.80 | 138.07 | 36.80 |
| 18.80 | 138.67 | 36.82 | 23.90 | 138.00 | 36.82 |
| 18.90 | 138.73 | 36.71 | 24.00 | 138.00 | 36.83 |
| 19.00 | 138.87 | 36.70 | 24.10 | 137.91 | 36.85 |
| 19.30 | 138.93 | 36.69 | 24.20 | 137.90 | 36.89 |
| 19.40 | 138.93 | 36.68 | 24.30 | 137.89 | 36.87 |
| 19.50 | 139.00 | 36.71 | 24.40 | 137.88 | 36.91 |
| 19.60 | 138.93 | 36.68 | | | |
| 19.70 | 138.93 | 36.70 | | | |
| 19.80 | 138.93 | 36.77 | | | |
| 19.90 | 138.87 | 36.80 | | | |

Table A-14

Experimental Results for Run # 03-LT

Initial Oxidation Temperature = 116 deg. C
Average System Pressure = 520 Psig.

Inlet Gas Composition = 40 % O₂
Average Gas Flow Rate = 80 cc/min

| Time, Hrs. | Temp. °C | O ₂ Conc. % | Time, Hrs. | Temp. °C | O ₂ Conc. % |
|------------|----------|------------------------|------------|----------|------------------------|
| 0.000 | 119.875 | 36.875 | 0.425 | 125.267 | 32.400 |
| 0.004 | 120.111 | 36.844 | 0.429 | 125.333 | 32.433 |
| 0.104 | 120.300 | 28.830 | 0.433 | 125.333 | 32.440 |
| 0.121 | 120.545 | 28.673 | 0.438 | 125.267 | 32.420 |
| 0.125 | 120.833 | 29.033 | 0.442 | 125.200 | 32.440 |
| 0.208 | 121.000 | 31.969 | 0.446 | 125.200 | 32.447 |
| 0.233 | 121.214 | 31.957 | 0.450 | 125.133 | 32.520 |
| 0.237 | 121.400 | 31.967 | 0.454 | 125.133 | 32.580 |
| 0.254 | 121.800 | 31.660 | 0.458 | 125.200 | 32.560 |
| 0.258 | 122.267 | 31.313 | 0.463 | 125.200 | 32.593 |
| 0.308 | 122.533 | 31.573 | 0.467 | 125.200 | 32.633 |
| 0.312 | 122.867 | 31.787 | 0.471 | 125.133 | 32.627 |
| 0.317 | 123.267 | 32.000 | 0.483 | 125.067 | 32.633 |
| 0.321 | 123.400 | 32.073 | 0.488 | 125.067 | 32.687 |
| 0.325 | 123.533 | 32.107 | 0.492 | 125.067 | 32.720 |
| 0.329 | 123.667 | 32.207 | 0.496 | 125.133 | 32.753 |
| 0.333 | 123.800 | 32.233 | 0.500 | 125.133 | 32.707 |
| 0.338 | 123.867 | 32.267 | 0.504 | 125.200 | 32.693 |
| 0.342 | 124.000 | 32.260 | 0.508 | 125.333 | 32.707 |
| 0.346 | 124.067 | 32.247 | 0.512 | 125.467 | 32.720 |
| 0.350 | 124.200 | 32.253 | 0.517 | 125.600 | 32.687 |
| 0.354 | 124.267 | 32.267 | 0.521 | 125.667 | 32.700 |
| 0.358 | 124.333 | 32.307 | 0.525 | 125.733 | 32.687 |
| 0.363 | 124.333 | 32.313 | 0.529 | 125.800 | 32.687 |
| 0.367 | 124.400 | 32.360 | 0.533 | 125.933 | 32.673 |
| 0.371 | 124.533 | 32.360 | 0.542 | 126.133 | 32.633 |
| 0.375 | 124.667 | 32.353 | 0.550 | 126.267 | 32.653 |
| 0.379 | 124.667 | 32.367 | 0.558 | 126.400 | 32.613 |
| 0.383 | 124.800 | 32.313 | 0.567 | 126.533 | 32.580 |
| 0.387 | 124.867 | 32.293 | 0.575 | 126.600 | 32.573 |
| 0.392 | 124.933 | 32.340 | 0.583 | 126.667 | 32.540 |
| 0.396 | 125.000 | 32.373 | 0.592 | 126.800 | 32.573 |
| 0.400 | 125.133 | 32.407 | 0.600 | 126.933 | 32.573 |
| 0.404 | 125.200 | 32.420 | 0.608 | 126.933 | 32.607 |
| 0.408 | 125.267 | 32.427 | 0.617 | 126.933 | 32.607 |
| 0.412 | 125.267 | 32.413 | 0.625 | 126.933 | 32.660 |
| 0.417 | 125.267 | 32.433 | 0.633 | 127.000 | 32.633 |

Table A-14 Contd.

| Time, Hrs. | Temp. °C | O ₂ Conc. % | Time, Hrs. | Temp. °C | O ₂ Conc. % |
|------------|----------|------------------------|------------|----------|------------------------|
| 0.642 | 127.133 | 32.600 | 1.033 | 128.133 | 32.407 |
| 0.658 | 127.133 | 32.600 | 1.042 | 128.200 | 32.433 |
| 0.667 | 127.133 | 32.587 | 1.050 | 128.333 | 32.407 |
| 0.675 | 127.067 | 32.567 | 1.058 | 128.333 | 32.447 |
| 0.683 | 127.067 | 32.567 | 1.067 | 128.333 | 32.400 |
| 0.692 | 127.067 | 32.620 | 1.083 | 128.400 | 32.360 |
| 0.700 | 127.133 | 32.607 | 1.100 | 128.400 | 32.287 |
| 0.708 | 127.200 | 32.640 | 1.117 | 128.400 | 32.307 |
| 0.717 | 127.200 | 32.647 | 1.133 | 128.400 | 32.233 |
| 0.725 | 127.200 | 32.607 | 1.150 | 128.467 | 32.173 |
| 0.733 | 127.133 | 32.620 | 1.167 | 128.533 | 32.240 |
| 0.742 | 127.133 | 32.580 | 1.183 | 128.600 | 32.253 |
| 0.750 | 127.267 | 32.567 | 1.200 | 128.600 | 32.200 |
| 0.767 | 127.333 | 32.533 | 1.217 | 128.533 | 32.147 |
| 0.775 | 127.400 | 32.507 | 1.233 | 128.600 | 32.147 |
| 0.783 | 127.467 | 32.540 | 1.250 | 128.667 | 32.087 |
| 0.792 | 127.533 | 32.580 | 1.267 | 128.600 | 32.053 |
| 0.800 | 127.600 | 32.620 | 1.283 | 128.667 | 32.053 |
| 0.808 | 127.733 | 32.647 | 1.300 | 128.800 | 32.013 |
| 0.817 | 127.800 | 32.640 | 1.317 | 128.867 | 31.987 |
| 0.825 | 127.867 | 32.660 | 1.333 | 128.933 | 31.953 |
| 0.833 | 127.867 | 32.620 | 1.350 | 129.067 | 32.000 |
| 0.842 | 127.933 | 32.560 | 1.367 | 129.200 | 31.993 |
| 0.850 | 128.067 | 32.600 | 1.383 | 129.400 | 32.007 |
| 0.858 | 128.200 | 32.600 | 1.400 | 129.467 | 32.047 |
| 0.867 | 128.267 | 32.573 | 1.417 | 129.533 | 31.980 |
| 0.875 | 128.400 | 32.593 | 1.433 | 129.600 | 31.967 |
| 0.883 | 128.333 | 32.533 | 1.450 | 129.733 | 31.960 |
| 0.892 | 128.333 | 32.533 | 1.467 | 129.933 | 31.987 |
| 0.900 | 128.333 | 32.533 | 1.483 | 129.933 | 31.967 |
| 0.908 | 128.267 | 32.507 | 1.500 | 130.000 | 31.993 |
| 0.917 | 128.200 | 32.453 | 1.517 | 130.133 | 31.993 |
| 0.925 | 128.200 | 32.387 | 1.533 | 130.200 | 32.020 |
| 0.933 | 128.200 | 32.393 | 1.550 | 130.200 | 32.013 |
| 0.942 | 128.200 | 32.413 | 1.567 | 130.200 | 31.980 |
| 0.950 | 128.200 | 32.393 | 1.583 | 130.200 | 31.960 |
| 0.958 | 128.200 | 32.380 | 1.600 | 130.200 | 31.933 |
| 0.967 | 128.200 | 32.393 | 1.617 | 130.200 | 31.933 |
| 0.975 | 128.133 | 32.387 | 1.633 | 130.133 | 31.987 |
| 0.983 | 128.067 | 32.360 | 1.650 | 130.133 | 31.953 |
| 0.992 | 128.067 | 32.353 | 1.667 | 130.133 | 31.967 |
| 1.000 | 128.000 | 32.340 | 1.683 | 130.200 | 31.967 |
| 1.008 | 128.067 | 32.380 | 1.700 | 130.200 | 31.993 |
| 1.025 | 128.133 | 32.393 | 1.717 | 130.200 | 31.940 |

Table A-14 Contd.

| Time, Hrs. | Temp. °C | O ₂ Conc. % | Time, Hrs. | Temp. °C | O ₂ Conc. % |
|------------|----------|------------------------|------------|----------|------------------------|
| 1.733 | 130.200 | 31.920 | 3.550 | 130.867 | 33.920 |
| 1.750 | 130.267 | 31.927 | 3.600 | 130.867 | 34.007 |
| 1.767 | 130.333 | 31.920 | 3.650 | 130.867 | 34.013 |
| 1.783 | 130.267 | 31.907 | 3.700 | 130.800 | 34.093 |
| 1.800 | 130.267 | 31.873 | 3.750 | 130.800 | 34.147 |
| 1.817 | 130.267 | 31.927 | 3.800 | 130.800 | 34.247 |
| 1.833 | 130.267 | 31.960 | 3.850 | 130.800 | 34.247 |
| 1.850 | 130.333 | 31.960 | 3.900 | 130.733 | 34.287 |
| 1.867 | 130.333 | 32.000 | 3.950 | 130.867 | 34.307 |
| 1.883 | 130.333 | 31.940 | 4.000 | 130.933 | 34.353 |
| 1.900 | 130.333 | 31.940 | 4.050 | 131.000 | 34.360 |
| 1.917 | 130.333 | 31.967 | 4.100 | 130.933 | 34.373 |
| 1.933 | 130.267 | 31.993 | 4.150 | 130.933 | 34.333 |
| 1.950 | 130.333 | 32.013 | 4.200 | 130.933 | 34.413 |
| 2.050 | 130.333 | 32.293 | 4.300 | 131.000 | 34.453 |
| 2.100 | 130.400 | 32.313 | 4.350 | 130.933 | 34.467 |
| 2.150 | 130.467 | 32.353 | 4.400 | 130.933 | 34.500 |
| 2.250 | 130.467 | 32.420 | 4.450 | 131.000 | 34.527 |
| 2.300 | 130.533 | 32.520 | 4.500 | 131.000 | 34.540 |
| 2.350 | 130.533 | 32.580 | 4.550 | 131.000 | 34.520 |
| 2.400 | 130.600 | 32.640 | 4.600 | 131.000 | 34.527 |
| 2.450 | 130.600 | 32.700 | 4.650 | 131.133 | 34.553 |
| 2.500 | 130.600 | 32.787 | 4.700 | 131.067 | 34.613 |
| 2.550 | 130.533 | 32.840 | 4.750 | 131.133 | 34.627 |
| 2.600 | 130.600 | 32.933 | 4.800 | 131.133 | 34.667 |
| 2.650 | 130.667 | 32.927 | 4.850 | 131.200 | 34.673 |
| 2.700 | 130.667 | 32.980 | 4.900 | 131.200 | 34.733 |
| 2.750 | 130.667 | 33.013 | 4.950 | 131.200 | 34.793 |
| 2.800 | 130.667 | 33.093 | 5.000 | 131.200 | 34.827 |
| 2.850 | 130.733 | 33.093 | 5.050 | 131.133 | 34.860 |
| 2.900 | 130.733 | 33.140 | 5.100 | 131.267 | 34.873 |
| 2.950 | 130.733 | 33.187 | 5.150 | 131.267 | 34.940 |
| 3.000 | 130.733 | 33.240 | 5.200 | 131.333 | 34.953 |
| 3.050 | 130.733 | 33.220 | 5.250 | 131.333 | 34.973 |
| 3.100 | 130.800 | 33.340 | 5.300 | 131.333 | 34.987 |
| 3.150 | 130.800 | 33.400 | 5.350 | 131.333 | 35.027 |
| 3.200 | 130.800 | 33.447 | 5.400 | 131.333 | 35.027 |
| 3.250 | 130.800 | 33.460 | 5.450 | 131.400 | 35.000 |
| 3.300 | 130.800 | 33.533 | 5.500 | 131.333 | 35.013 |
| 3.350 | 130.867 | 33.580 | 5.550 | 131.400 | 35.020 |
| 3.400 | 130.867 | 33.700 | 5.600 | 131.333 | 35.040 |
| 3.450 | 130.867 | 33.733 | 5.650 | 131.400 | 35.020 |
| 3.500 | 130.867 | 33.840 | 5.700 | 131.400 | 34.980 |

Table A-14 Contd.

| Time, Hrs. | Temp. °C | O ₂ Conc. % |
|------------|----------|------------------------|
| 5.750 | 131.467 | 34.920 |
| 5.800 | 131.467 | 34.880 |
| 5.850 | 131.467 | 34.880 |
| 5.900 | 131.533 | 34.840 |
| 5.950 | 131.467 | 34.873 |
| 6.000 | 131.533 | 34.880 |
| 6.200 | 131.533 | 35.020 |
| 6.400 | 131.400 | 35.073 |
| 6.600 | 131.533 | 35.093 |
| 6.800 | 131.600 | 35.193 |
| 7.000 | 131.667 | 35.300 |
| 7.200 | 131.800 | 35.373 |
| 7.400 | 131.733 | 35.600 |
| 7.600 | 131.800 | 35.560 |
| 7.800 | 131.733 | 35.547 |
| 8.000 | 131.667 | 35.627 |
| 8.200 | 131.667 | 35.567 |
| 8.400 | 131.733 | 35.447 |
| 8.600 | 131.800 | 35.427 |
| 8.800 | 131.867 | 35.333 |
| 9.000 | 131.933 | 35.327 |
| 9.200 | 131.867 | 35.393 |
| 9.400 | 131.867 | 35.507 |
| 9.600 | 131.733 | 35.500 |
| 9.800 | 131.667 | 35.593 |
| 10.000 | 131.733 | 35.887 |
| 10.200 | 131.667 | 35.700 |
| 10.400 | 131.400 | 35.693 |
| 10.600 | 130.867 | 35.760 |
| 10.800 | 130.267 | 35.680 |
| 11.000 | 130.000 | 35.867 |
| 11.200 | 130.000 | 35.907 |
| 11.400 | 130.000 | 35.814 |
| 11.600 | 130.100 | 35.850 |
| 11.700 | 130.000 | 35.813 |

Table A-15

Experimental Results for Run # 04-LT

Initial Oxidation Temperature = 113 deg. C
Average System Pressure = 500-80 Psig.

Inlet Gas Composition = 40 % O₂
Average Gas Flow Rate = 80 cc/min

| Time, Hrs. | Temp. °C | O ₂ Conc. % | Time, Hrs. | Temp. °C | O ₂ Conc. % |
|------------|----------|------------------------|------------|----------|------------------------|
| 0.000 | 115.250 | 35.588 | 0.167 | 126.667 | 34.980 |
| 0.004 | 115.444 | 35.578 | 0.171 | 126.733 | 34.953 |
| 0.008 | 115.700 | 35.570 | 0.175 | 126.800 | 34.933 |
| 0.013 | 116.091 | 35.555 | 0.179 | 126.867 | 34.913 |
| 0.017 | 116.500 | 35.550 | 0.183 | 126.933 | 34.900 |
| 0.021 | 116.846 | 35.539 | 0.188 | 127.000 | 34.887 |
| 0.025 | 117.143 | 35.536 | 0.192 | 127.000 | 34.867 |
| 0.029 | 117.400 | 35.533 | 0.196 | 127.000 | 34.847 |
| 0.033 | 117.867 | 35.520 | 0.200 | 127.000 | 34.827 |
| 0.038 | 118.467 | 35.507 | 0.204 | 126.933 | 34.807 |
| 0.042 | 119.133 | 35.493 | 0.208 | 126.933 | 34.787 |
| 0.046 | 119.600 | 35.480 | 0.213 | 127.067 | 34.760 |
| 0.050 | 120.067 | 35.460 | 0.217 | 127.133 | 34.747 |
| 0.054 | 120.667 | 35.447 | 0.221 | 127.133 | 34.727 |
| 0.058 | 121.200 | 35.433 | 0.225 | 127.133 | 34.707 |
| 0.062 | 121.733 | 35.427 | 0.229 | 127.133 | 34.687 |
| 0.067 | 122.200 | 35.413 | 0.233 | 127.133 | 34.680 |
| 0.071 | 122.667 | 35.400 | 0.237 | 127.200 | 34.667 |
| 0.075 | 122.933 | 35.393 | 0.242 | 127.267 | 34.647 |
| 0.079 | 123.133 | 35.380 | 0.246 | 127.267 | 34.633 |
| 0.083 | 123.333 | 35.373 | 0.250 | 127.267 | 34.647 |
| 0.087 | 123.600 | 35.347 | 0.254 | 127.333 | 33.520 |
| 0.092 | 123.933 | 35.320 | 0.258 | 127.467 | 31.800 |
| 0.096 | 124.200 | 35.307 | 0.262 | 127.600 | 29.920 |
| 0.100 | 124.333 | 35.287 | 0.267 | 127.733 | 27.967 |
| 0.104 | 124.467 | 35.260 | 0.271 | 127.867 | 25.973 |
| 0.108 | 124.667 | 35.240 | 0.275 | 127.933 | 23.953 |
| 0.112 | 124.867 | 35.227 | 0.279 | 128.000 | 21.907 |
| 0.117 | 125.000 | 35.207 | 0.283 | 128.067 | 19.840 |
| 0.121 | 125.133 | 35.180 | 0.287 | 128.200 | 17.767 |
| 0.125 | 125.267 | 35.153 | 0.292 | 128.333 | 15.680 |
| 0.129 | 125.467 | 35.133 | 0.296 | 128.400 | 13.573 |
| 0.133 | 125.600 | 35.113 | 0.300 | 128.467 | 11.467 |
| 0.138 | 125.800 | 35.093 | 0.304 | 128.533 | 9.360 |
| 0.142 | 126.067 | 35.073 | 0.308 | 128.667 | 7.247 |
| 0.146 | 126.267 | 35.053 | 0.312 | 128.800 | 5.100 |
| 0.150 | 126.333 | 35.047 | 0.317 | 128.867 | 4.107 |
| 0.154 | 126.400 | 35.033 | 0.321 | 128.867 | 3.700 |
| 0.158 | 126.467 | 35.013 | 0.325 | 128.867 | 3.447 |
| 0.162 | 126.600 | 34.993 | 0.329 | 128.867 | 3.267 |

Table A-15 Contd.

| Time, Hrs. | Temp. °C | O ₂ Conc. % | Time, Hrs. | Temp. °C | O ₂ Conc. % |
|------------|----------|------------------------|------------|----------|------------------------|
| 0.333 | 128.800 | 3.120 | 0.512 | 127.000 | 2.200 |
| 0.338 | 128.800 | 3.007 | 0.517 | 127.067 | 2.187 |
| 0.342 | 128.800 | 2.913 | 0.521 | 127.067 | 2.173 |
| 0.346 | 128.867 | 2.847 | 0.525 | 127.067 | 2.160 |
| 0.350 | 128.667 | 2.793 | 0.529 | 127.133 | 2.140 |
| 0.354 | 128.533 | 2.747 | 0.533 | 127.133 | 2.120 |
| 0.358 | 128.467 | 2.713 | 0.538 | 127.133 | 2.100 |
| 0.363 | 128.333 | 2.680 | 0.542 | 127.133 | 2.073 |
| 0.367 | 128.200 | 2.653 | 0.546 | 127.200 | 2.047 |
| 0.371 | 128.067 | 2.627 | 0.550 | 127.200 | 2.040 |
| 0.375 | 127.933 | 2.607 | 0.554 | 127.200 | 2.053 |
| 0.379 | 127.800 | 2.587 | 0.558 | 127.200 | 2.047 |
| 0.383 | 127.667 | 2.567 | 0.562 | 127.200 | 2.040 |
| 0.387 | 127.533 | 2.547 | 0.567 | 127.267 | 2.020 |
| 0.392 | 127.400 | 2.527 | 0.571 | 127.267 | 2.013 |
| 0.396 | 127.333 | 2.520 | 0.575 | 127.200 | 1.993 |
| 0.400 | 127.200 | 2.513 | 0.579 | 127.267 | 1.980 |
| 0.404 | 127.000 | 2.500 | 0.583 | 127.267 | 1.967 |
| 0.408 | 126.800 | 2.487 | 0.587 | 127.267 | 1.953 |
| 0.412 | 126.867 | 2.473 | 0.592 | 127.200 | 1.947 |
| 0.417 | 126.867 | 2.467 | 0.596 | 127.133 | 1.940 |
| 0.421 | 126.867 | 2.460 | 0.600 | 127.200 | 1.933 |
| 0.425 | 126.867 | 2.453 | 0.604 | 127.200 | 1.933 |
| 0.429 | 126.867 | 2.440 | 0.608 | 127.133 | 1.940 |
| 0.433 | 126.867 | 2.427 | 0.613 | 127.133 | 1.933 |
| 0.438 | 126.867 | 2.413 | 0.617 | 127.133 | 1.913 |
| 0.442 | 126.867 | 2.400 | 0.621 | 127.133 | 1.907 |
| 0.446 | 126.867 | 2.387 | 0.625 | 127.200 | 1.913 |
| 0.450 | 126.867 | 2.380 | 0.629 | 127.133 | 1.927 |
| 0.454 | 126.800 | 2.373 | 0.633 | 127.133 | 1.933 |
| 0.458 | 126.800 | 2.360 | 0.637 | 127.267 | 1.953 |
| 0.463 | 126.800 | 2.347 | 0.642 | 127.200 | 1.960 |
| 0.467 | 126.867 | 2.340 | 0.646 | 127.200 | 1.967 |
| 0.471 | 127.000 | 2.333 | 0.650 | 127.200 | 1.973 |
| 0.475 | 127.000 | 2.327 | 0.654 | 127.200 | 1.980 |
| 0.479 | 127.000 | 2.320 | 0.658 | 127.267 | 1.987 |
| 0.483 | 127.000 | 2.313 | 0.663 | 127.200 | 2.000 |
| 0.488 | 127.000 | 2.300 | 0.667 | 127.200 | 2.007 |
| 0.492 | 127.000 | 2.287 | 0.671 | 127.133 | 2.000 |
| 0.496 | 127.000 | 2.273 | 0.675 | 127.133 | 2.007 |
| 0.500 | 127.000 | 2.253 | 0.679 | 127.133 | 2.013 |
| 0.504 | 127.000 | 2.233 | 0.683 | 127.133 | 2.027 |
| 0.508 | 127.000 | 2.213 | 0.688 | 127.067 | 2.027 |

Table A-15 Contd.

| Time, Hrs. | Temp. °C | O ₂ Conc. % | Time, Hrs. | Temp. °C | O ₂ Conc. % |
|------------|----------|------------------------|------------|----------|------------------------|
| 0.692 | 127.067 | 2.033 | 0.879 | 125.733 | 2.107 |
| 0.696 | 127.067 | 2.033 | 0.883 | 125.667 | 2.120 |
| 0.700 | 127.000 | 2.033 | 0.887 | 125.667 | 2.120 |
| 0.704 | 127.000 | 2.040 | 0.892 | 125.667 | 2.120 |
| 0.708 | 127.000 | 2.040 | 0.896 | 125.733 | 2.127 |
| 0.712 | 127.000 | 2.040 | 0.900 | 125.667 | 2.133 |
| 0.717 | 127.000 | 2.040 | 0.904 | 125.600 | 2.140 |
| 0.721 | 126.933 | 2.047 | 0.908 | 125.600 | 2.147 |
| 0.725 | 127.000 | 2.040 | 0.913 | 125.733 | 2.160 |
| 0.729 | 127.000 | 2.047 | 0.917 | 125.800 | 2.173 |
| 0.733 | 127.067 | 2.060 | 0.921 | 125.733 | 2.187 |
| 0.738 | 127.067 | 2.060 | 0.925 | 125.733 | 2.200 |
| 0.742 | 127.067 | 2.060 | 0.929 | 125.733 | 2.200 |
| 0.746 | 127.067 | 2.053 | 0.933 | 125.667 | 2.207 |
| 0.754 | 127.067 | 2.060 | 0.938 | 125.667 | 2.220 |
| 0.758 | 127.067 | 2.060 | 0.942 | 125.733 | 2.233 |
| 0.762 | 127.000 | 2.053 | 0.958 | 125.733 | 2.300 |
| 0.767 | 126.933 | 2.047 | 0.962 | 125.800 | 2.313 |
| 0.771 | 126.933 | 2.053 | 0.967 | 125.867 | 2.327 |
| 0.775 | 126.800 | 2.053 | 0.971 | 125.867 | 2.340 |
| 0.779 | 126.800 | 2.060 | 0.975 | 125.733 | 2.353 |
| 0.783 | 126.733 | 2.053 | 0.979 | 125.733 | 2.373 |
| 0.788 | 126.600 | 2.060 | 0.983 | 125.800 | 2.387 |
| 0.792 | 126.533 | 2.053 | 0.988 | 125.800 | 2.407 |
| 0.796 | 126.400 | 2.047 | 0.992 | 125.867 | 2.427 |
| 0.800 | 126.333 | 2.047 | 0.996 | 125.933 | 2.447 |
| 0.804 | 126.267 | 2.047 | 1.000 | 125.933 | 2.460 |
| 0.808 | 126.200 | 2.053 | 1.004 | 125.800 | 2.480 |
| 0.812 | 126.067 | 2.047 | 1.008 | 125.733 | 2.487 |
| 0.817 | 126.000 | 2.047 | 1.013 | 125.800 | 2.500 |
| 0.821 | 125.933 | 2.047 | 1.017 | 125.733 | 2.500 |
| 0.825 | 125.933 | 2.053 | 1.021 | 125.733 | 2.507 |
| 0.829 | 125.933 | 2.060 | 1.025 | 125.667 | 2.527 |
| 0.833 | 125.800 | 2.060 | 1.029 | 125.667 | 2.547 |
| 0.837 | 125.867 | 2.067 | 1.033 | 125.667 | 2.560 |
| 0.842 | 125.800 | 2.067 | 1.038 | 125.733 | 2.573 |
| 0.846 | 125.800 | 2.073 | 1.042 | 125.733 | 2.580 |
| 0.850 | 125.733 | 2.073 | 1.046 | 125.667 | 2.600 |
| 0.854 | 125.667 | 2.080 | 1.050 | 125.667 | 2.600 |
| 0.858 | 125.733 | 2.087 | 1.054 | 125.600 | 2.620 |
| 0.863 | 125.733 | 2.087 | 1.058 | 125.533 | 2.633 |
| 0.867 | 125.667 | 2.093 | 1.062 | 125.533 | 2.660 |
| 0.871 | 125.667 | 2.100 | 1.067 | 125.600 | 2.680 |
| 0.875 | 125.733 | 2.107 | 1.071 | 125.667 | 2.707 |

Table A-15 Contd.

| Time, Hrs. | Temp. °C | O ₂ Conc. % | Time, Hrs. | Temp. °C | O ₂ Conc. % |
|------------|----------|------------------------|------------|----------|------------------------|
| 1.075 | 125.667 | 2.727 | 1.254 | 125.867 | 3.693 |
| 1.079 | 125.667 | 2.760 | 1.258 | 125.867 | 3.720 |
| 1.083 | 125.667 | 2.787 | 1.263 | 125.800 | 3.740 |
| 1.087 | 125.733 | 2.800 | 1.267 | 125.800 | 3.760 |
| 1.092 | 125.733 | 2.820 | 1.271 | 125.867 | 3.793 |
| 1.096 | 125.733 | 2.847 | 1.275 | 125.800 | 3.820 |
| 1.100 | 125.800 | 2.867 | 1.279 | 125.867 | 3.833 |
| 1.104 | 125.800 | 2.880 | 1.283 | 125.800 | 3.853 |
| 1.108 | 125.867 | 2.900 | 1.288 | 125.867 | 3.880 |
| 1.112 | 125.867 | 2.933 | 1.292 | 125.867 | 3.907 |
| 1.117 | 125.933 | 2.953 | 1.296 | 125.800 | 3.933 |
| 1.121 | 126.000 | 2.973 | 1.300 | 125.733 | 3.953 |
| 1.125 | 126.000 | 3.007 | 1.304 | 125.733 | 3.980 |
| 1.129 | 125.933 | 3.020 | 1.308 | 125.733 | 4.000 |
| 1.133 | 125.933 | 3.040 | 1.312 | 125.667 | 4.020 |
| 1.138 | 125.933 | 3.060 | 1.317 | 125.600 | 4.040 |
| 1.142 | 126.000 | 3.080 | 1.321 | 125.533 | 4.053 |
| 1.146 | 125.933 | 3.100 | 1.325 | 125.600 | 4.073 |
| 1.150 | 125.933 | 3.120 | 1.329 | 125.600 | 4.100 |
| 1.154 | 125.933 | 3.133 | 1.333 | 125.533 | 4.120 |
| 1.158 | 125.867 | 3.153 | 1.337 | 125.533 | 4.107 |
| 1.163 | 125.800 | 3.180 | 1.354 | 125.467 | 3.993 |
| 1.167 | 125.733 | 3.213 | 1.358 | 125.467 | 3.887 |
| 1.171 | 125.733 | 3.227 | 1.362 | 125.467 | 3.787 |
| 1.175 | 125.667 | 3.240 | 1.367 | 125.400 | 3.680 |
| 1.179 | 125.667 | 3.260 | 1.371 | 125.333 | 3.580 |
| 1.183 | 125.667 | 3.280 | 1.375 | 125.400 | 3.473 |
| 1.188 | 125.600 | 3.280 | 1.379 | 125.400 | 3.367 |
| 1.192 | 125.600 | 3.300 | 1.383 | 125.467 | 3.273 |
| 1.196 | 125.600 | 3.320 | 1.388 | 125.467 | 3.167 |
| 1.200 | 125.600 | 3.347 | 1.392 | 125.467 | 3.060 |
| 1.204 | 125.600 | 3.373 | 1.396 | 125.533 | 2.953 |
| 1.208 | 125.600 | 3.393 | 1.400 | 125.600 | 2.887 |
| 1.212 | 125.600 | 3.420 | 1.404 | 125.600 | 2.780 |
| 1.217 | 125.533 | 3.453 | 1.408 | 125.600 | 2.687 |
| 1.221 | 125.600 | 3.480 | 1.413 | 125.667 | 2.660 |
| 1.225 | 125.533 | 3.500 | 1.417 | 125.733 | 2.667 |
| 1.229 | 125.600 | 3.520 | 1.421 | 125.733 | 2.687 |
| 1.233 | 125.667 | 3.547 | 1.425 | 125.800 | 2.713 |
| 1.237 | 125.733 | 3.573 | 1.429 | 125.867 | 2.733 |
| 1.242 | 125.733 | 3.600 | 1.433 | 125.867 | 2.753 |
| 1.246 | 125.733 | 3.627 | 1.438 | 125.800 | 2.773 |
| 1.250 | 125.800 | 3.660 | 1.442 | 125.867 | 2.793 |

Table A-15 Contd.

| Time, Hrs. | Temp. °C | O ₂ Conc. % | Time, Hrs. | Temp. °C | O ₂ Conc. % |
|------------|----------|------------------------|------------|----------|------------------------|
| 1.446 | 125.867 | 2.813 | 1.625 | 125.533 | 3.327 |
| 1.450 | 125.867 | 2.840 | 1.629 | 125.600 | 3.340 |
| 1.454 | 125.867 | 2.867 | 1.633 | 125.600 | 3.347 |
| 1.458 | 125.867 | 2.887 | 1.638 | 125.533 | 3.353 |
| 1.462 | 125.867 | 2.900 | 1.642 | 125.600 | 3.360 |
| 1.467 | 125.867 | 2.913 | 1.646 | 125.667 | 3.373 |
| 1.471 | 125.800 | 2.933 | 1.650 | 125.733 | 3.387 |
| 1.475 | 125.800 | 2.960 | 1.654 | 125.733 | 3.400 |
| 1.479 | 125.800 | 2.980 | 1.658 | 125.733 | 3.413 |
| 1.483 | 125.800 | 3.000 | 1.663 | 125.733 | 3.420 |
| 1.487 | 125.800 | 3.013 | 1.667 | 125.800 | 3.433 |
| 1.492 | 125.733 | 3.027 | 1.671 | 125.733 | 3.453 |
| 1.496 | 125.800 | 3.047 | 1.675 | 125.733 | 3.473 |
| 1.500 | 125.800 | 3.060 | 1.679 | 125.667 | 3.507 |
| 1.504 | 125.733 | 3.080 | 1.683 | 125.667 | 3.533 |
| 1.508 | 125.667 | 3.100 | 1.688 | 125.600 | 3.560 |
| 1.513 | 125.600 | 3.113 | 1.692 | 125.600 | 3.587 |
| 1.517 | 125.600 | 3.120 | 1.696 | 125.600 | 3.613 |
| 1.521 | 125.600 | 3.133 | 1.700 | 125.667 | 3.640 |
| 1.525 | 125.600 | 3.147 | 1.704 | 125.600 | 3.667 |
| 1.529 | 125.533 | 3.160 | 1.708 | 125.600 | 3.687 |
| 1.533 | 125.600 | 3.167 | 1.712 | 125.467 | 3.707 |
| 1.538 | 125.600 | 3.173 | 1.717 | 125.467 | 3.733 |
| 1.542 | 125.600 | 3.180 | 1.721 | 125.400 | 3.760 |
| 1.546 | 125.600 | 3.187 | 1.725 | 125.333 | 3.787 |
| 1.550 | 125.533 | 3.193 | 1.729 | 125.333 | 3.807 |
| 1.554 | 125.533 | 3.200 | 1.733 | 125.267 | 3.827 |
| 1.558 | 125.533 | 3.207 | 1.746 | 125.333 | 3.853 |
| 1.562 | 125.600 | 3.220 | 1.750 | 125.400 | 3.860 |
| 1.567 | 125.600 | 3.220 | 1.754 | 125.400 | 3.873 |
| 1.571 | 125.667 | 3.220 | 1.758 | 125.333 | 3.887 |
| 1.575 | 125.733 | 3.227 | 1.763 | 125.333 | 3.900 |
| 1.579 | 125.667 | 3.233 | 1.767 | 125.267 | 3.913 |
| 1.583 | 125.600 | 3.240 | 1.771 | 125.133 | 3.927 |
| 1.587 | 125.533 | 3.247 | 1.775 | 125.267 | 3.947 |
| 1.592 | 125.600 | 3.253 | 1.779 | 125.133 | 3.953 |
| 1.596 | 125.533 | 3.260 | 1.783 | 125.133 | 3.967 |
| 1.600 | 125.467 | 3.273 | 1.788 | 125.267 | 3.980 |
| 1.604 | 125.400 | 3.287 | 1.792 | 125.267 | 3.993 |
| 1.608 | 125.400 | 3.293 | 1.796 | 125.400 | 4.007 |
| 1.612 | 125.467 | 3.307 | 1.800 | 125.400 | 4.027 |
| 1.617 | 125.533 | 3.313 | 1.804 | 125.333 | 4.040 |
| 1.621 | 125.533 | 3.320 | 1.808 | 125.267 | 4.053 |

Table A-15 Contd.

| Time, Hrs. | Temp. °C | O ₂ Conc. % | Time, Hrs. | Temp. °C | O ₂ Conc. % |
|------------|----------|------------------------|------------|----------|------------------------|
| 1.812 | 125.200 | 4.067 | 1.996 | 123.933 | 4.867 |
| 1.817 | 125.200 | 4.080 | 2.000 | 124.000 | 4.880 |
| 1.821 | 125.133 | 4.093 | 2.004 | 124.000 | 4.887 |
| 1.825 | 125.000 | 4.107 | 2.008 | 124.000 | 4.893 |
| 1.829 | 125.133 | 4.127 | 2.013 | 124.000 | 4.907 |
| 1.833 | 125.267 | 4.147 | 2.017 | 124.000 | 4.920 |
| 1.837 | 125.133 | 4.153 | 2.021 | 124.000 | 4.927 |
| 1.842 | 125.133 | 4.167 | 2.025 | 123.933 | 4.933 |
| 1.846 | 125.133 | 4.180 | 2.029 | 123.933 | 4.953 |
| 1.850 | 125.067 | 4.193 | 2.033 | 124.000 | 4.960 |
| 1.854 | 125.067 | 4.213 | 2.037 | 124.000 | 4.967 |
| 1.858 | 124.933 | 4.240 | 2.042 | 123.933 | 4.973 |
| 1.862 | 124.800 | 4.273 | 2.046 | 123.933 | 4.980 |
| 1.867 | 124.733 | 4.307 | 2.050 | 123.933 | 4.993 |
| 1.871 | 124.667 | 4.333 | 2.054 | 123.933 | 5.007 |
| 1.875 | 124.600 | 4.373 | 2.058 | 123.933 | 5.020 |
| 1.879 | 124.467 | 4.407 | 2.062 | 123.867 | 5.027 |
| 1.883 | 124.467 | 4.440 | 2.067 | 123.867 | 5.047 |
| 1.888 | 124.400 | 4.473 | 2.071 | 123.867 | 5.053 |
| 1.892 | 124.267 | 4.500 | 2.075 | 123.867 | 5.067 |
| 1.896 | 124.133 | 4.533 | 2.079 | 123.867 | 5.073 |
| 1.900 | 124.133 | 4.573 | 2.083 | 123.867 | 5.080 |
| 1.904 | 124.133 | 4.613 | 2.088 | 123.933 | 5.087 |
| 1.908 | 124.133 | 4.647 | 2.092 | 124.000 | 5.093 |
| 1.913 | 124.133 | 4.680 | 2.096 | 124.000 | 5.107 |
| 1.917 | 124.000 | 4.707 | 2.100 | 124.000 | 5.120 |
| 1.921 | 124.000 | 4.727 | 2.104 | 124.067 | 5.140 |
| 1.925 | 124.000 | 4.740 | 2.108 | 124.133 | 5.153 |
| 1.929 | 124.000 | 4.753 | 2.112 | 124.200 | 5.160 |
| 1.933 | 124.000 | 4.767 | 2.117 | 124.267 | 5.173 |
| 1.938 | 124.000 | 4.773 | 2.121 | 124.267 | 5.187 |
| 1.942 | 124.000 | 4.780 | 2.125 | 124.267 | 5.193 |
| 1.946 | 124.000 | 4.793 | 2.133 | 124.333 | 5.220 |
| 1.950 | 124.067 | 4.800 | 2.138 | 124.400 | 5.233 |
| 1.954 | 124.067 | 4.807 | 2.142 | 124.400 | 5.253 |
| 1.958 | 124.067 | 4.813 | 2.146 | 124.400 | 5.287 |
| 1.962 | 124.067 | 4.820 | 2.150 | 124.400 | 5.320 |
| 1.967 | 124.067 | 4.820 | 2.154 | 124.333 | 5.360 |
| 1.971 | 124.000 | 4.827 | 2.158 | 124.400 | 5.400 |
| 1.975 | 123.933 | 4.833 | 2.162 | 124.400 | 5.447 |
| 1.979 | 123.933 | 4.840 | 2.167 | 124.400 | 5.493 |
| 1.983 | 123.933 | 4.847 | 2.171 | 124.333 | 5.540 |
| 1.987 | 123.933 | 4.853 | 2.175 | 124.267 | 5.593 |
| 1.992 | 123.933 | 4.860 | 2.179 | 124.200 | 5.647 |

Table A-15 Contd.

| Time, Hrs. | Temp. °C | O ₂ Conc. % | Time, Hrs. | Temp. °C | O ₂ Conc. % |
|------------|----------|------------------------|------------|----------|------------------------|
| 2.183 | 124.200 | 5.707 | 2.371 | 124.267 | 7.487 |
| 2.188 | 124.200 | 5.767 | 2.375 | 124.267 | 7.507 |
| 2.192 | 124.067 | 5.827 | 2.379 | 124.267 | 7.527 |
| 2.196 | 124.067 | 5.887 | 2.396 | 124.200 | 7.627 |
| 2.200 | 124.000 | 5.947 | 2.400 | 124.200 | 7.647 |
| 2.204 | 124.000 | 6.000 | 2.404 | 124.200 | 7.667 |
| 2.208 | 124.000 | 6.047 | 2.408 | 124.133 | 7.693 |
| 2.213 | 124.000 | 6.093 | 2.412 | 124.067 | 7.720 |
| 2.217 | 124.000 | 6.133 | 2.417 | 124.067 | 7.747 |
| 2.221 | 123.933 | 6.173 | 2.421 | 124.133 | 7.780 |
| 2.225 | 123.933 | 6.207 | 2.425 | 124.067 | 7.813 |
| 2.229 | 123.933 | 6.233 | 2.429 | 124.067 | 7.847 |
| 2.233 | 123.933 | 6.273 | 2.433 | 124.067 | 7.880 |
| 2.237 | 123.933 | 6.307 | 2.438 | 124.067 | 7.920 |
| 2.242 | 123.933 | 6.333 | 2.442 | 124.067 | 7.960 |
| 2.246 | 123.933 | 6.353 | 2.446 | 124.133 | 8.000 |
| 2.250 | 123.933 | 6.387 | 2.450 | 124.133 | 8.040 |
| 2.254 | 124.000 | 6.420 | 2.454 | 124.133 | 8.073 |
| 2.258 | 124.000 | 6.440 | 2.458 | 124.133 | 8.107 |
| 2.263 | 124.000 | 6.460 | 2.463 | 124.200 | 8.147 |
| 2.267 | 124.067 | 6.487 | 2.467 | 124.200 | 8.187 |
| 2.271 | 124.067 | 6.513 | 2.471 | 124.200 | 8.220 |
| 2.275 | 124.067 | 6.540 | 2.475 | 124.200 | 8.260 |
| 2.279 | 124.067 | 6.567 | 2.479 | 124.200 | 8.300 |
| 2.283 | 124.067 | 6.600 | 2.483 | 124.133 | 8.340 |
| 2.287 | 124.067 | 6.647 | 2.487 | 124.133 | 8.380 |
| 2.292 | 124.067 | 6.693 | 2.492 | 124.133 | 8.427 |
| 2.296 | 124.067 | 6.733 | 2.496 | 124.133 | 8.473 |
| 2.300 | 124.133 | 6.773 | 2.500 | 124.133 | 8.520 |
| 2.304 | 124.200 | 6.820 | 2.504 | 124.133 | 8.573 |
| 2.308 | 124.200 | 6.867 | 2.508 | 124.067 | 8.633 |
| 2.312 | 124.200 | 6.913 | 2.513 | 124.067 | 8.687 |
| 2.317 | 124.200 | 6.953 | 2.517 | 124.067 | 8.747 |
| 2.321 | 124.200 | 7.000 | 2.521 | 124.067 | 8.807 |
| 2.325 | 124.267 | 7.047 | 2.525 | 124.000 | 8.873 |
| 2.329 | 124.200 | 7.100 | 2.529 | 124.000 | 8.940 |
| 2.333 | 124.200 | 7.147 | 2.533 | 124.000 | 9.007 |
| 2.338 | 124.200 | 7.200 | 2.537 | 124.000 | 9.067 |
| 2.342 | 124.200 | 7.253 | 2.542 | 124.000 | 9.133 |
| 2.346 | 124.267 | 7.300 | 2.546 | 124.000 | 9.193 |
| 2.350 | 124.333 | 7.333 | 2.550 | 124.000 | 9.260 |
| 2.354 | 124.333 | 7.367 | 2.554 | 124.067 | 9.320 |
| 2.358 | 124.333 | 7.400 | 2.558 | 124.133 | 9.387 |
| 2.362 | 124.333 | 7.433 | 2.562 | 124.133 | 9.440 |
| 2.367 | 124.267 | 7.460 | 2.567 | 124.133 | 9.493 |

Table A-15 Contd.

| Time, Hr | Temp. °C | O ₂ Conc. % |
|----------|----------|------------------------|
| 2.571 | 124.133 | 9.540 |
| 2.575 | 124.133 | 9.587 |
| 2.579 | 124.133 | 9.627 |
| 2.583 | 124.133 | 9.667 |
| 2.588 | 124.133 | 9.700 |
| 2.592 | 124.133 | 9.727 |
| 2.596 | 124.133 | 9.753 |
| 2.600 | 124.133 | 9.780 |
| 2.604 | 124.133 | 9.793 |
| 2.608 | 124.133 | 9.813 |
| 2.612 | 124.133 | 9.827 |
| 2.617 | 124.067 | 9.840 |
| 2.621 | 124.000 | 9.847 |
| 2.625 | 124.067 | 9.860 |
| 2.629 | 124.067 | 9.867 |
| 2.633 | 124.133 | 9.873 |
| 2.638 | 124.133 | 9.880 |
| 2.642 | 124.133 | 9.880 |
| 2.646 | 124.133 | 9.887 |
| 2.650 | 124.133 | 9.880 |
| 2.654 | 124.133 | 9.880 |
| 2.658 | 124.133 | 9.880 |
| 2.662 | 124.133 | 9.873 |
| 2.667 | 124.133 | 9.880 |
| 2.671 | 124.143 | 9.879 |
| 2.675 | 124.154 | 9.877 |
| 2.679 | 124.167 | 9.875 |
| 2.683 | 124.182 | 9.873 |
| 2.688 | 124.100 | 9.870 |
| 2.692 | 124.111 | 9.867 |
| 2.696 | 124.000 | 9.863 |